Supporting Information

Synthesis of tetrazole derived organocatalysts via azido-Ugi reaction with cyclic ketimines

Olga I. Shmatova[a] and Valentine G. Nenajdenko[a*]

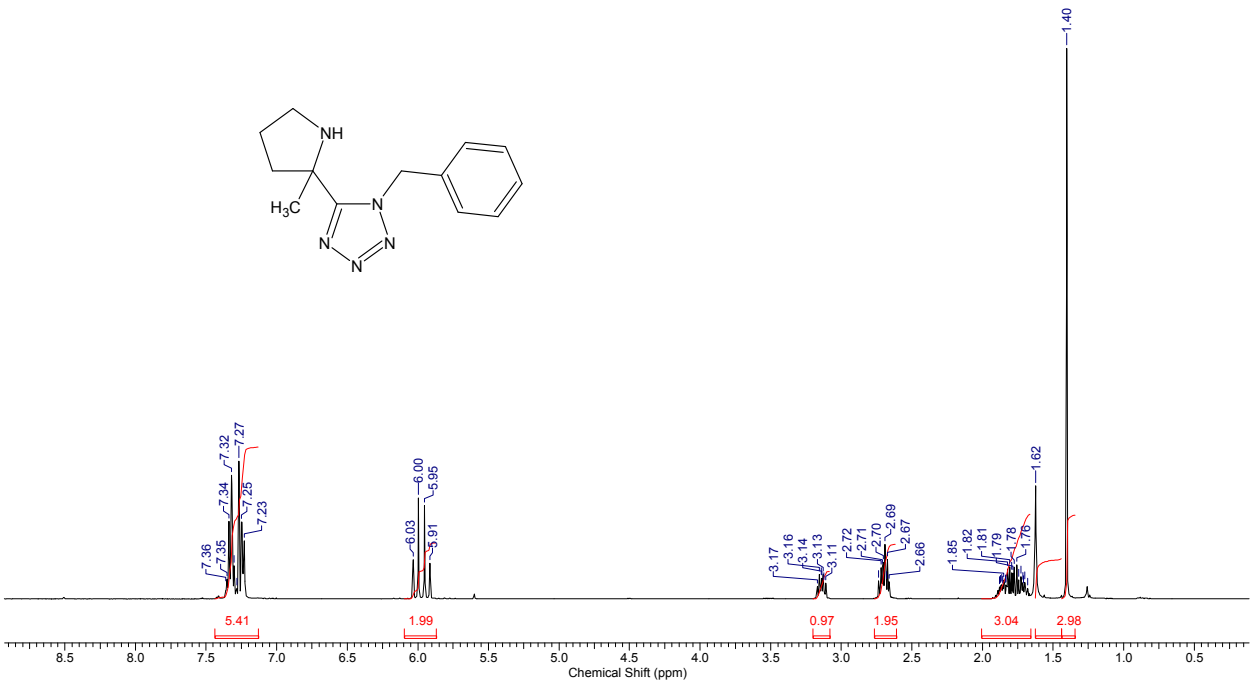
[a]*Department of Chemistry, Lomonosov Moscow State University, Moscow 119991, Russia*

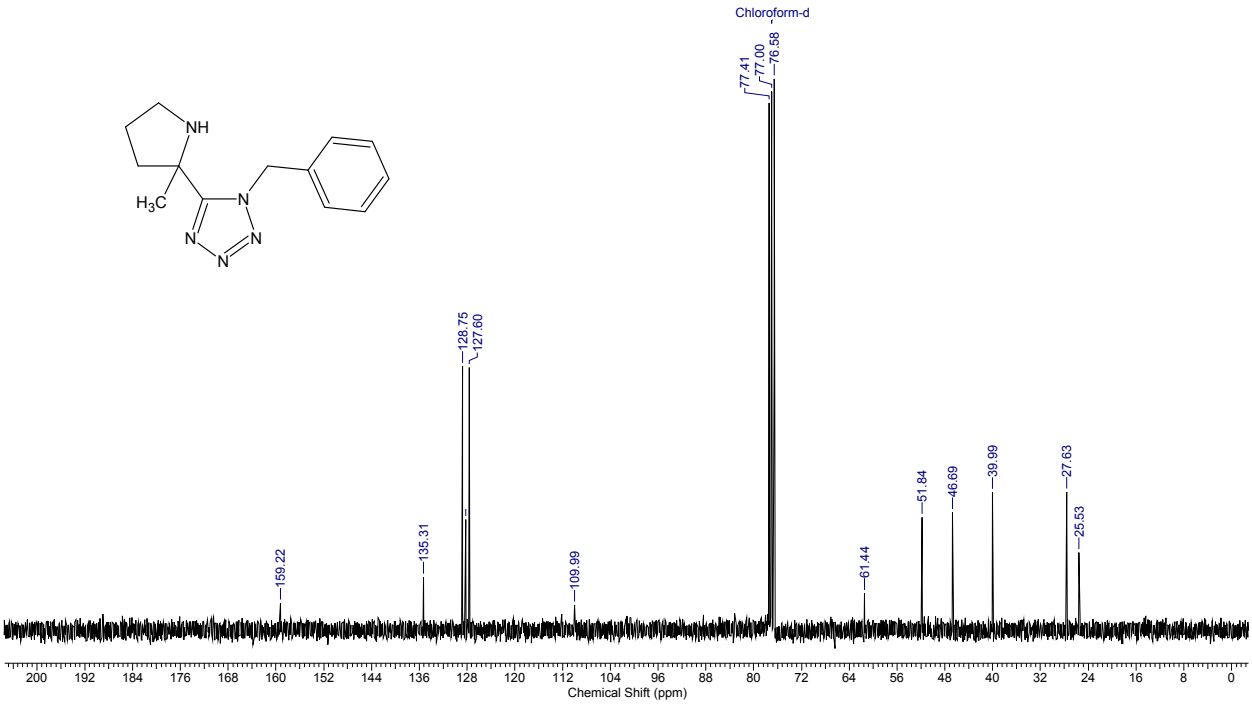
nen@acylium.chem.msu.ru (V. G. Nenajdenko)

Table of Content

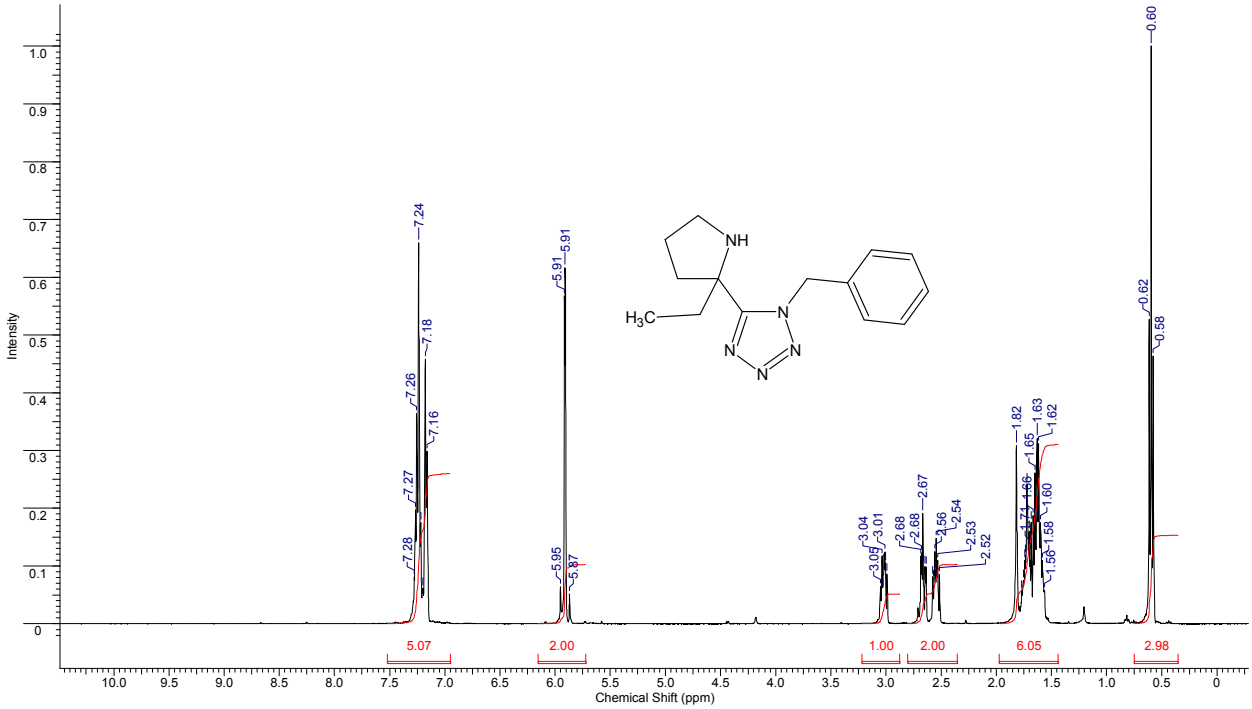
1. NMR spectra

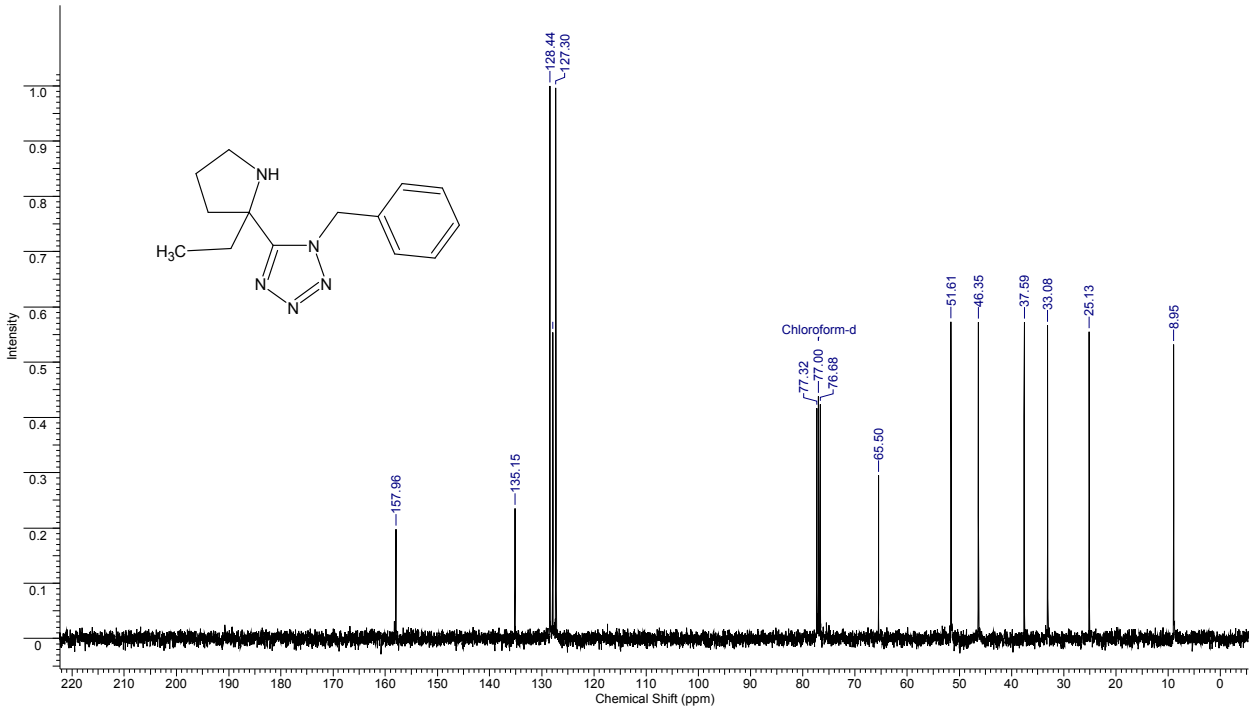
3a



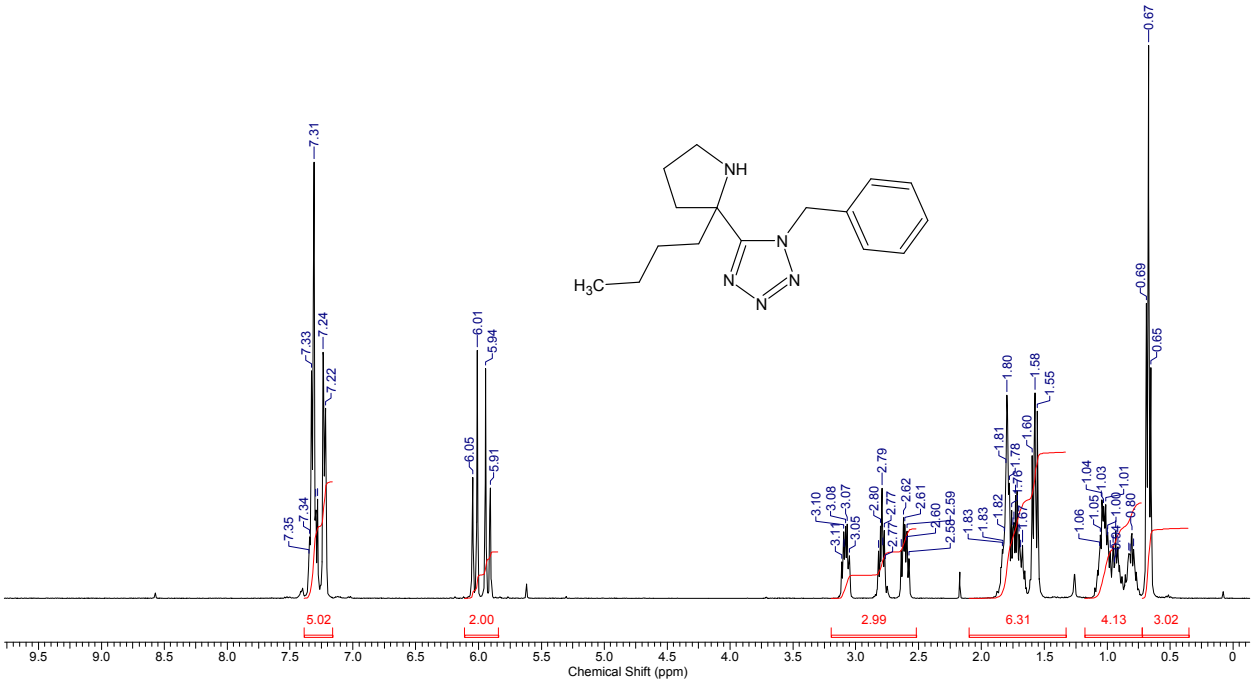

Chloroform-d
77.41
77.00
76.58
128.75
127.60
159.22
135.31
109.99
61.44
51.84
46.69
39.99
27.63
25.53
NH
H3C
N
N
N
N
Chemical Shift (ppm)

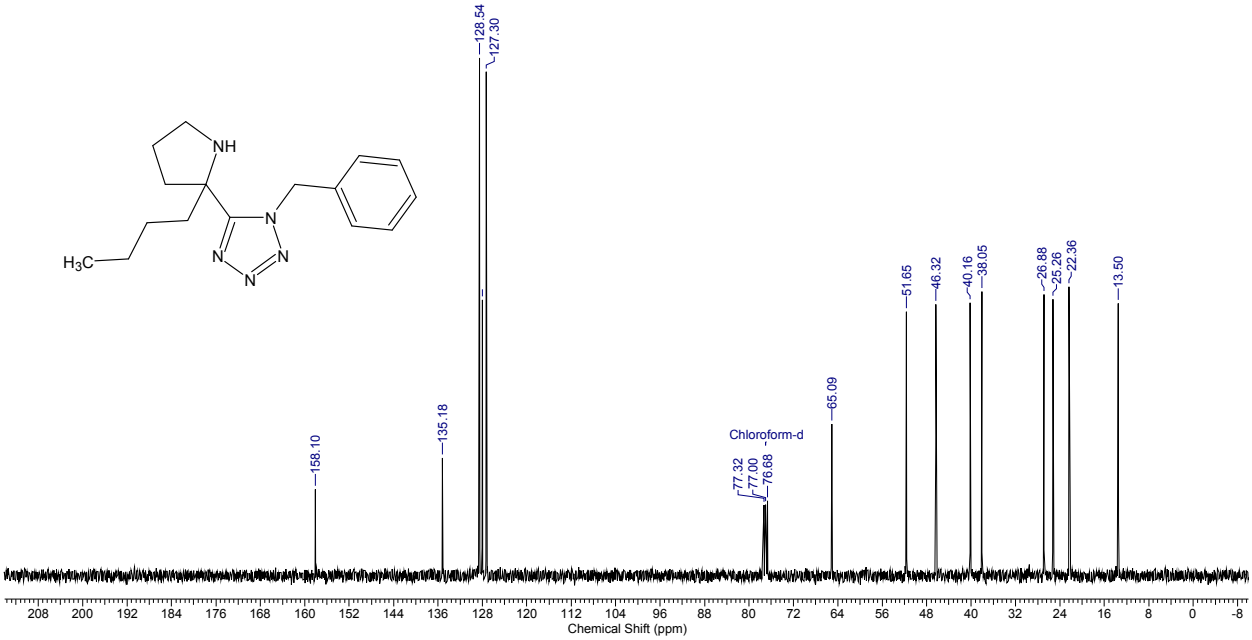
3b


Intensity
7.24
7.26
7.27
7.28
7.18
7.16
5.91
5.91
5.95
5.87
3.04
3.01
3.09
2.68
2.68
2.67
3.56
2.54
2.53
2.52
1.82
1.65
1.63
1.62
1.60
1.58
1.56
0.60
0.62
0.58
5.07
2.00
1.00
2.00
6.05
2.98
NH
H3C
N
N
N
N
Chemical Shift (ppm)

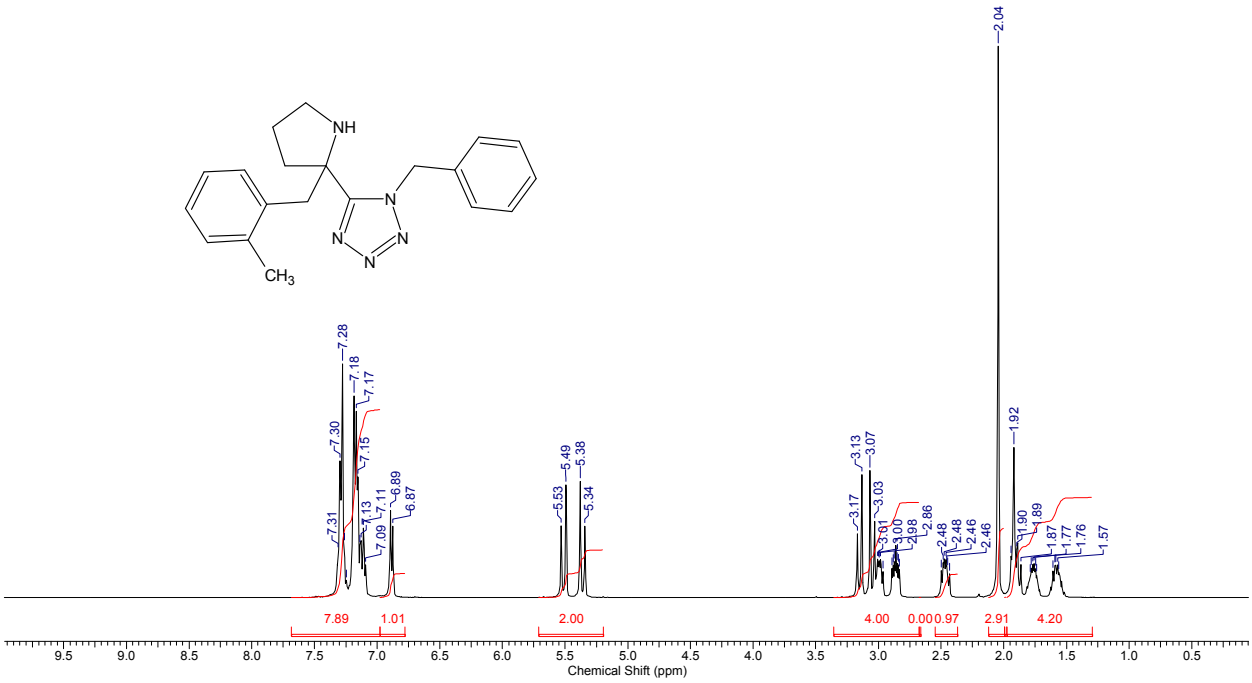

NH
H3C
N
N
N
N
128.44
127.30
157.96
135.15
Chloroform-d
77.32
77.00
76.68
65.50
51.61
46.35
37.59
33.08
25.13
8.95
Intensity
Chemical Shift (ppm)

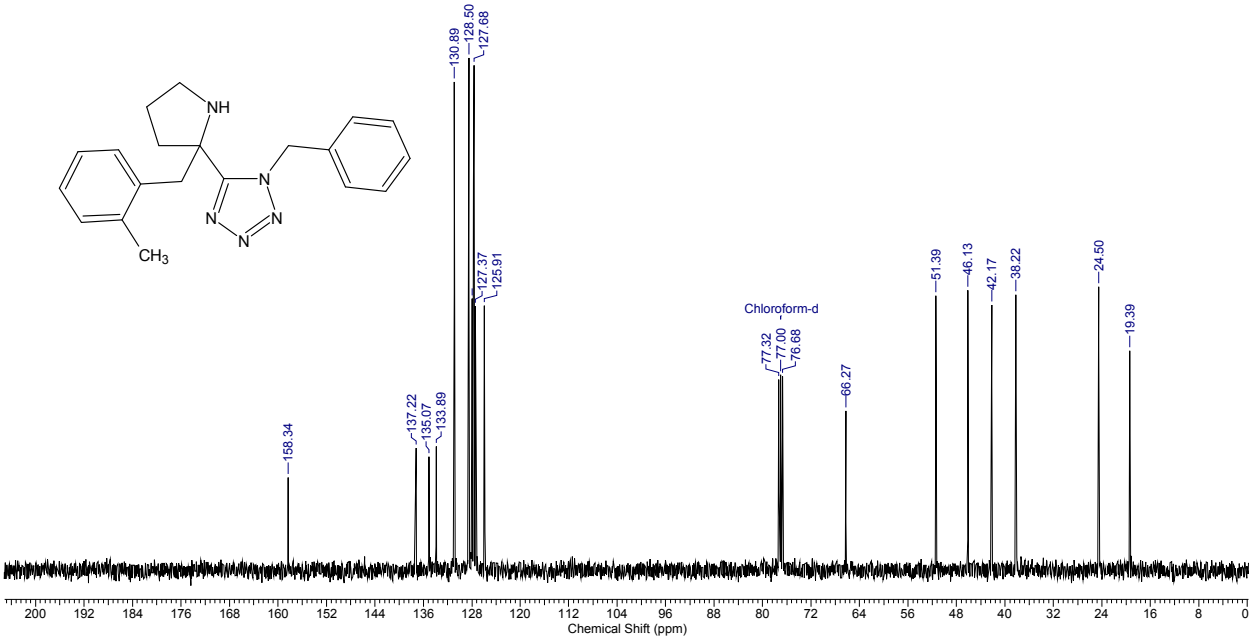
3c


NH
H3C
N
N
N
N
7.31
7.35
7.34
7.33
7.24
7.22
6.05
6.01
5.94
5.91
3.10
3.08
3.07
3.05
3.11
2.80
2.79
2.77
2.62
2.61
2.60
2.59
2.58
1.83
1.82
1.81
1.80
1.78
1.76
1.67
1.60
1.58
1.55
1.06
1.05
1.04
1.03
1.00
1.01
0.80
0.69
0.67
0.65
5.02
2.00
2.99
6.31
4.13
3.02
Chemical Shift (ppm)

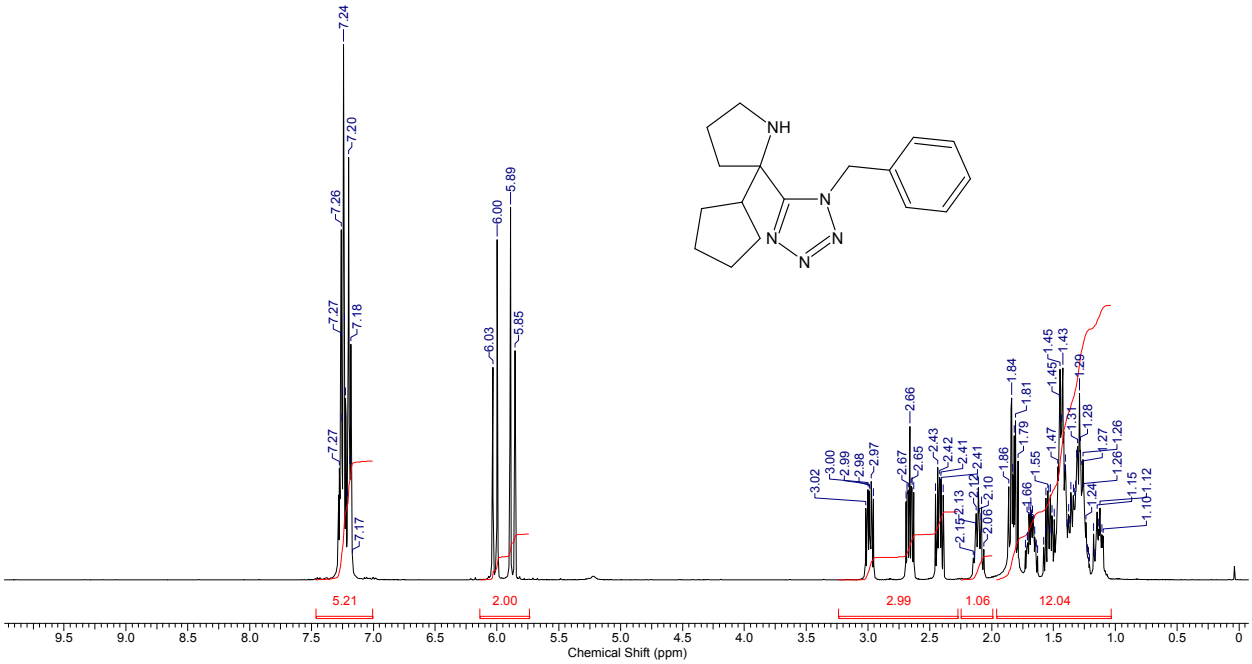

NH
N
N
N
N
H3C
128.54
127.30
158.10
135.18
Chloroform-d
77.32
77.00
76.68
65.09
51.65
46.32
40.16
38.05
26.88
25.26
22.36
13.50
Chemical Shift (ppm)

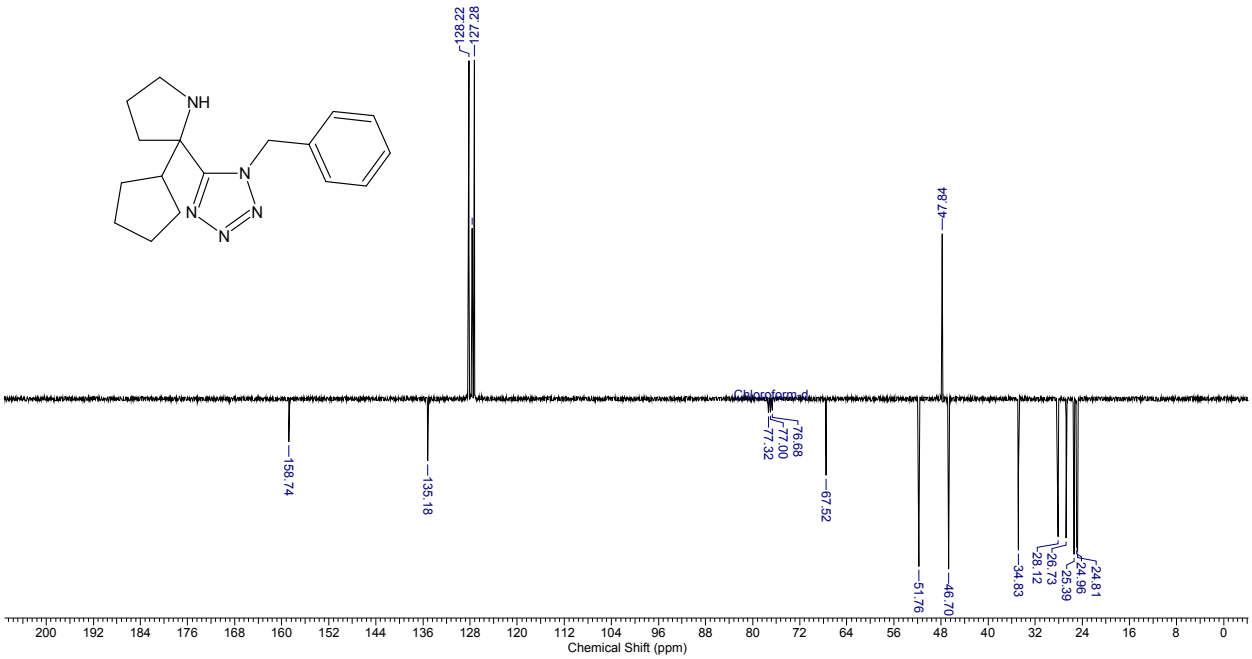
3d


NH
N
N
N
N
CH3
7.28
7.30
7.18
7.17
7.16
7.31
7.13
7.11
7.09
6.89
6.87
5.49
5.53
5.38
5.34
3.17
3.13
3.07
3.03
3.01
3.00
2.98
2.86
2.48
2.48
2.46
2.46
2.04
1.92
1.90
1.89
1.87
1.77
1.76
1.57
7.89
1.01
2.00
4.00
0.00
0.97
2.91
4.20
Chemical Shift (ppm)

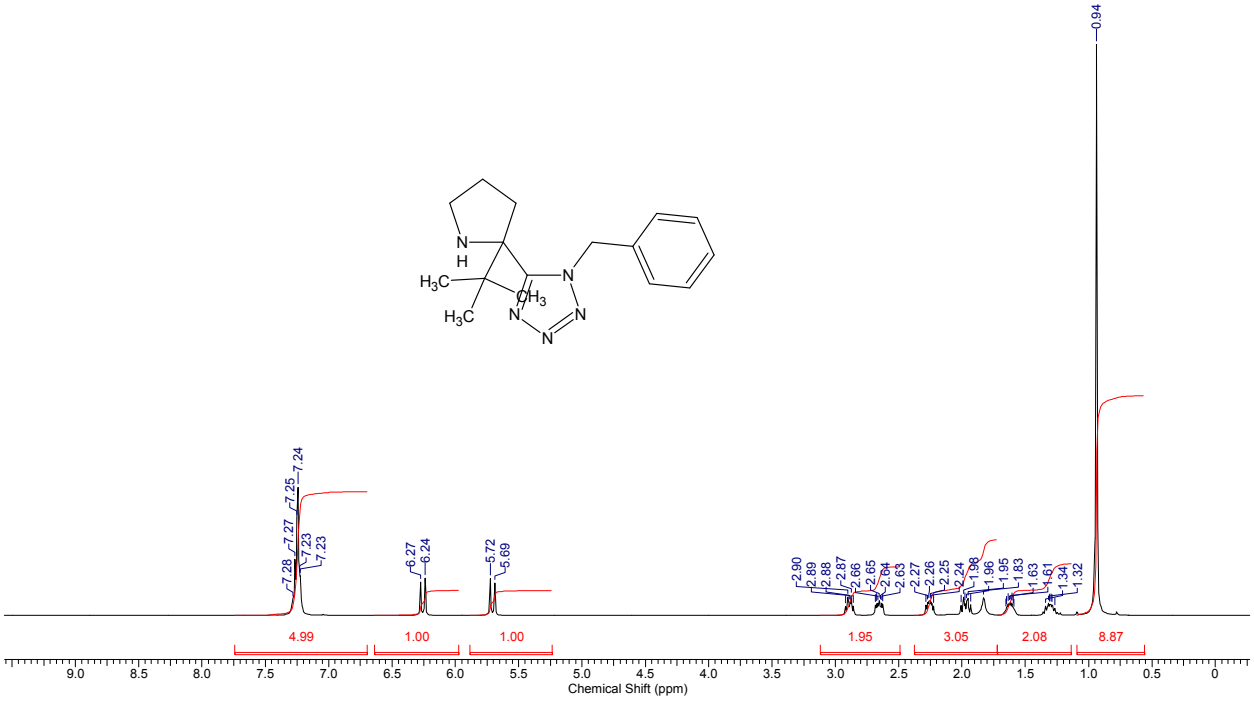

NH
N
N
N
N
CH3
130.89
128.50
127.68
127.37
125.91
158.34
137.22
135.07
133.89
Chloroform-d
77.32
77.00
76.68
66.27
51.39
46.13
42.17
38.22
24.50
19.39
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

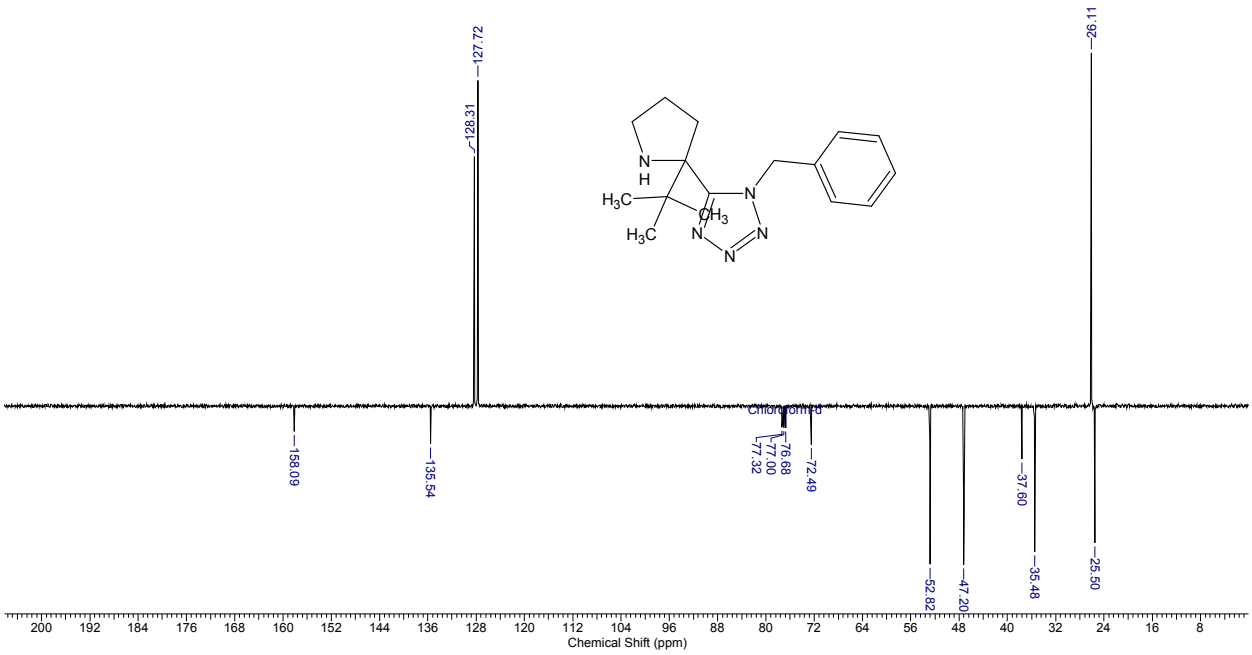
3e


NH
N
N
N
N
7.24
7.20
7.26
7.27
7.18
7.17
6.00
5.89
6.03
5.85
2.66
2.43
2.42
2.41
2.67
2.65
2.41
2.13
2.12
2.10
2.06
3.02
3.00
2.99
2.98
2.97
1.84
1.81
1.79
1.45
1.47
1.55
1.66
1.45
1.43
1.31
1.29
1.28
1.27
1.26
1.26
1.24
1.15
1.12
1.10
5.21
2.00
2.99
1.06
12.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
Chemical Shift (ppm)

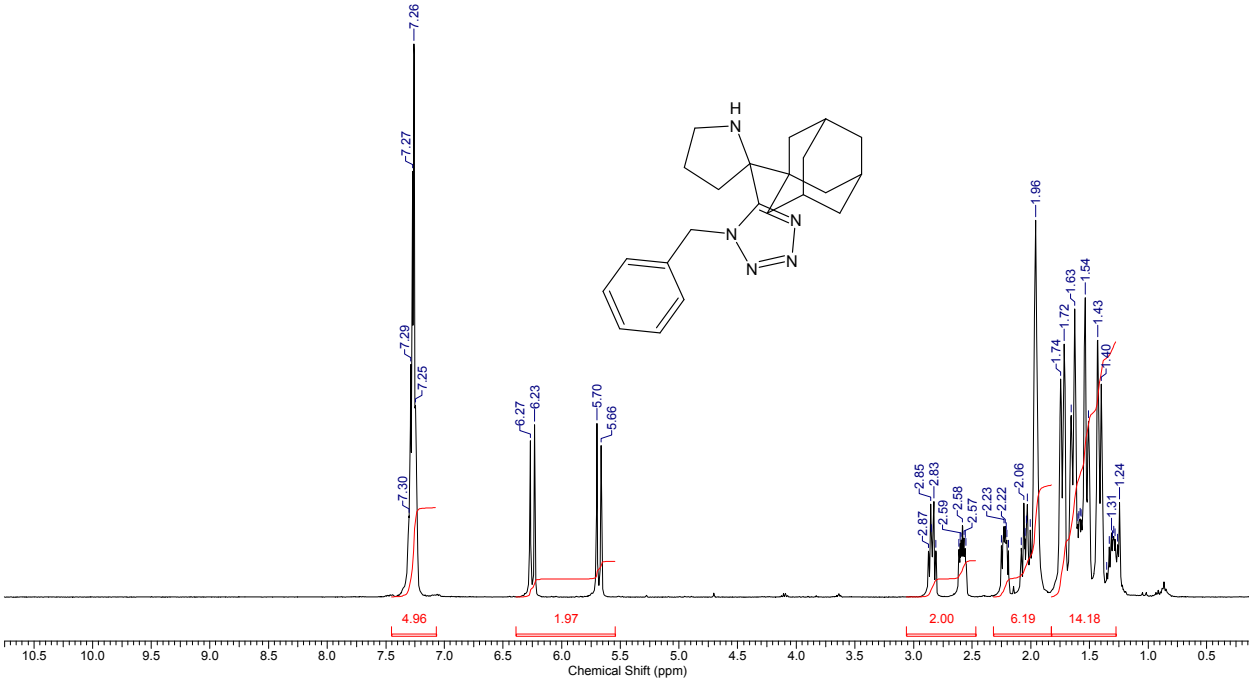

NH
N
N
N
N
128.22
127.28
47.84
Chloroform-d
77.32
77.00
76.68
158.74
135.18
67.52
51.76
46.70
34.83
28.12
26.73
25.39
24.96
24.81
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

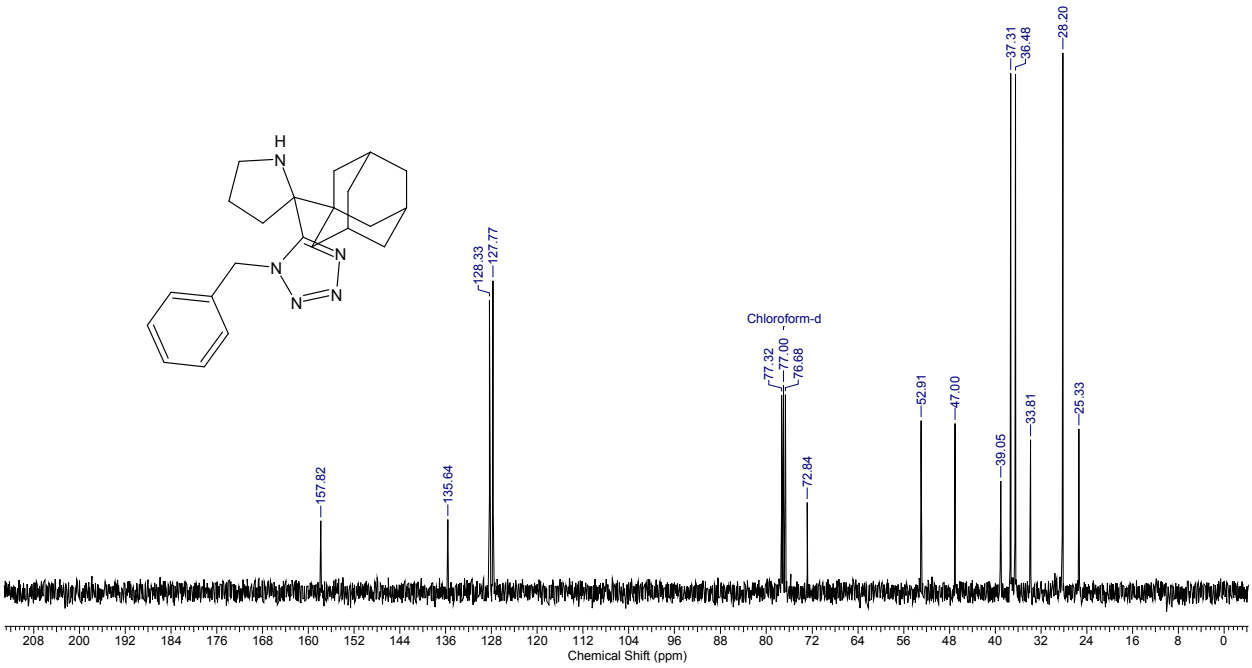
3f


N
H
H3C
CH3
H3C
N
N
N
9.60
7.28
7.27
7.25
7.24
7.23
7.23
6.27
6.24
5.72
5.69
2.90
2.89
2.88
2.87
2.66
2.65
2.64
2.63
2.27
2.26
2.25
2.24
1.98
1.96
1.95
1.83
1.63
1.61
1.34
1.32
4.99
1.00
1.00
1.95
3.05
2.08
8.87
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
Chemical Shift (ppm)

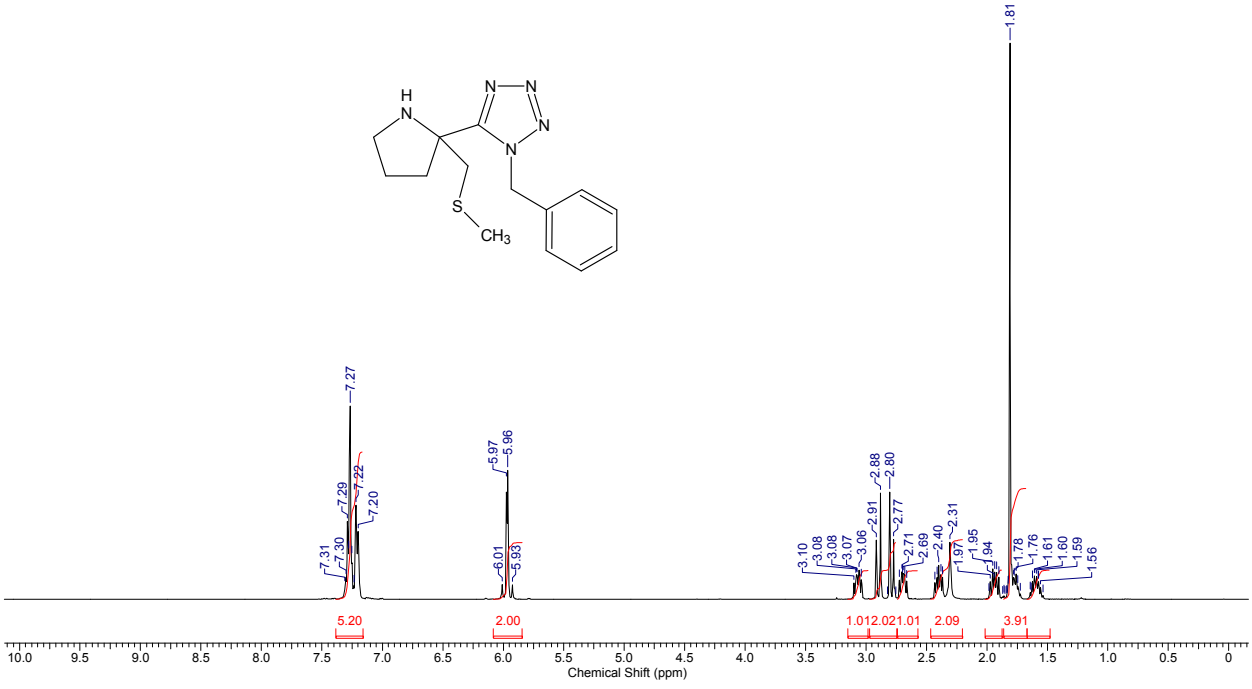

127.72
128.31
26.11
158.09
135.54
Chloroform-d
77.32
77.00
76.68
72.49
52.82
47.20
37.60
35.48
25.50
Chemical Shift (ppm)

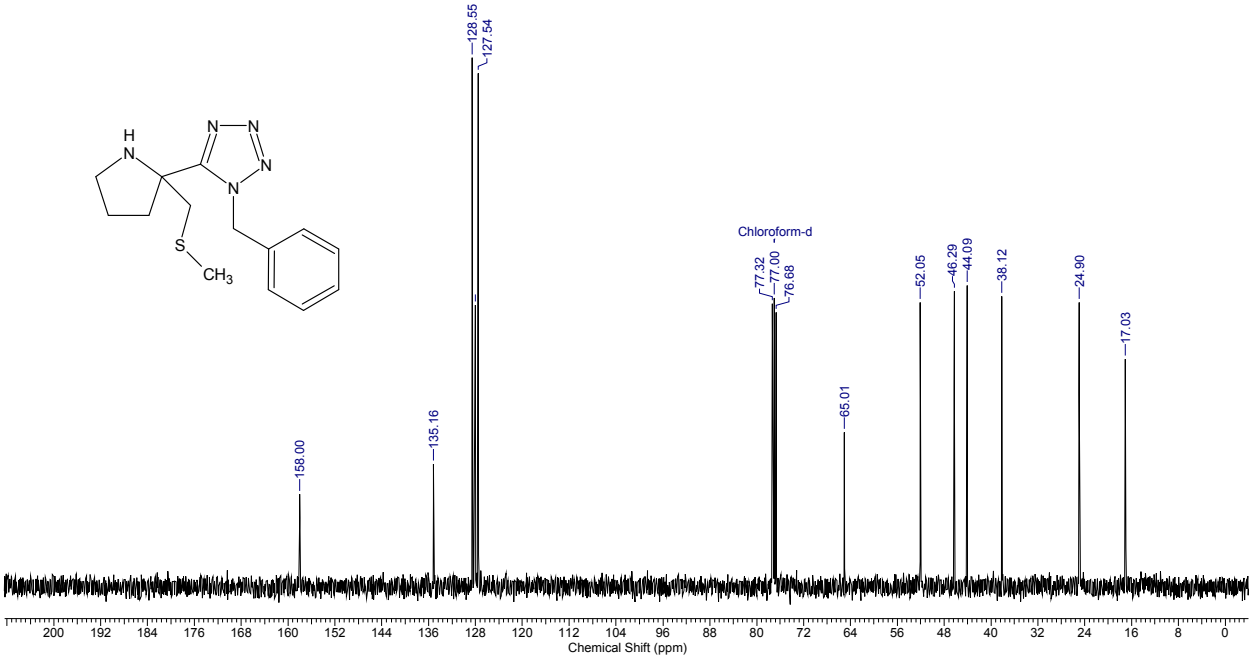
3g


7.26
7.27
7.29
7.25
7.30
6.27
6.23
5.70
5.66
2.85
2.83
2.87
2.59
2.58
2.57
2.23
2.22
2.06
1.96
1.74
1.72
1.63
1.54
1.43
1.40
1.31
1.24
4.96
1.97
2.00
6.19
14.18
Chemical Shift (ppm)

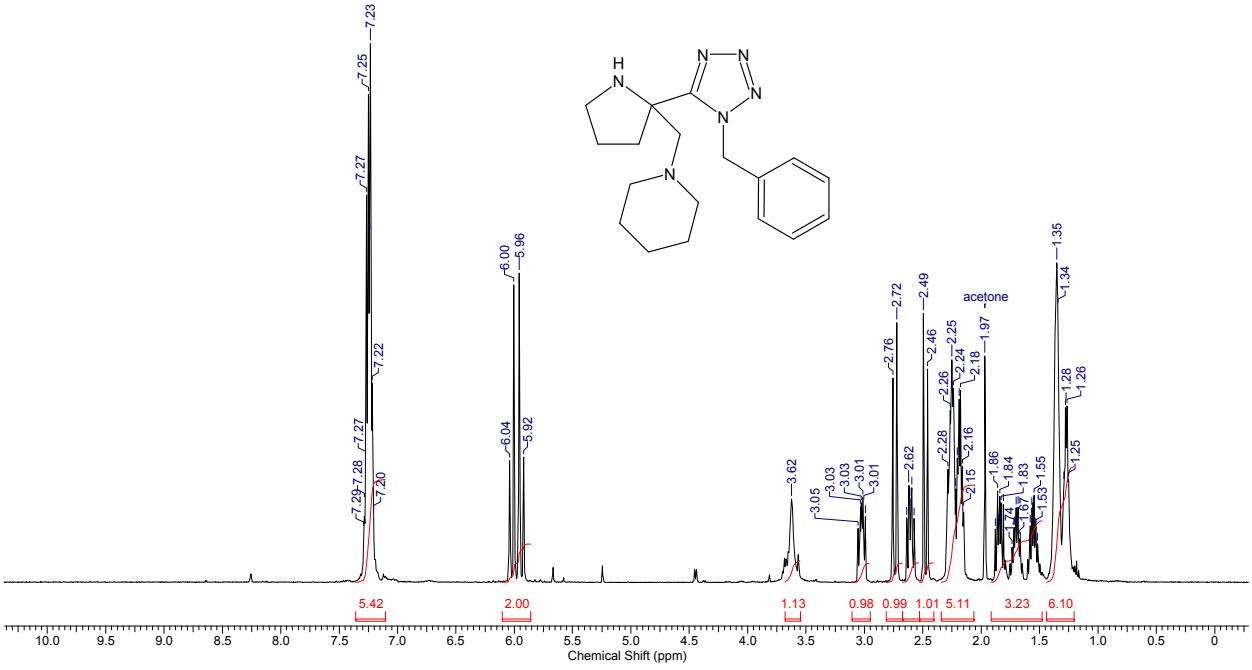

H
N
N
N
N
157.82
135.64
128.33
127.77
Chloroform-d
77.32
77.00
76.68
72.84
52.91
47.00
39.05
37.31
36.48
33.81
28.20
25.33
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

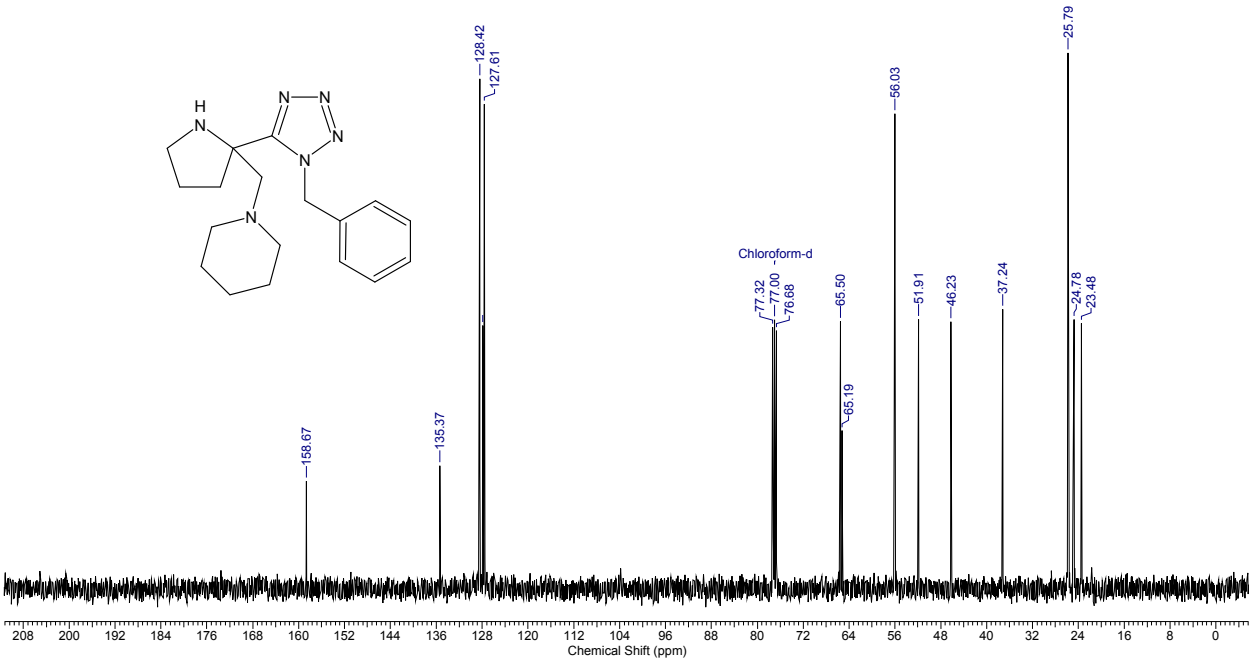
3i


H
N
N
N
N
S
CH3
1.81
7.27
7.31
7.30
7.29
7.22
7.20
5.97
5.96
6.01
5.93
2.88
2.80
2.91
2.77
2.71
2.69
2.40
2.31
1.97
1.95
1.94
3.10
3.08
3.08
3.07
3.06
1.78
1.76
1.61
1.60
1.59
1.56
5.20
2.00
1.01
2.02
1.01
2.09
3.91
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
Chemical Shift (ppm)

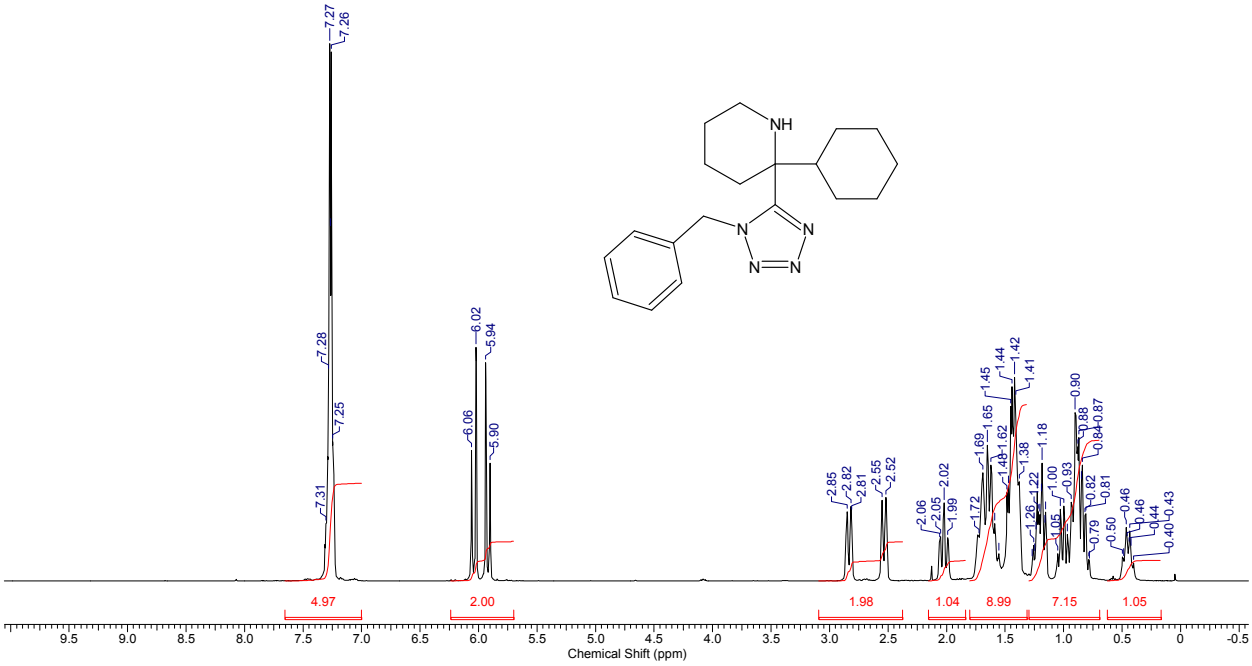

128.55
127.54
Chloroform-d
77.32
77.00
76.68
52.05
46.29
44.09
38.12
24.90
17.03
65.01
135.16
158.00
Chemical Shift (ppm)

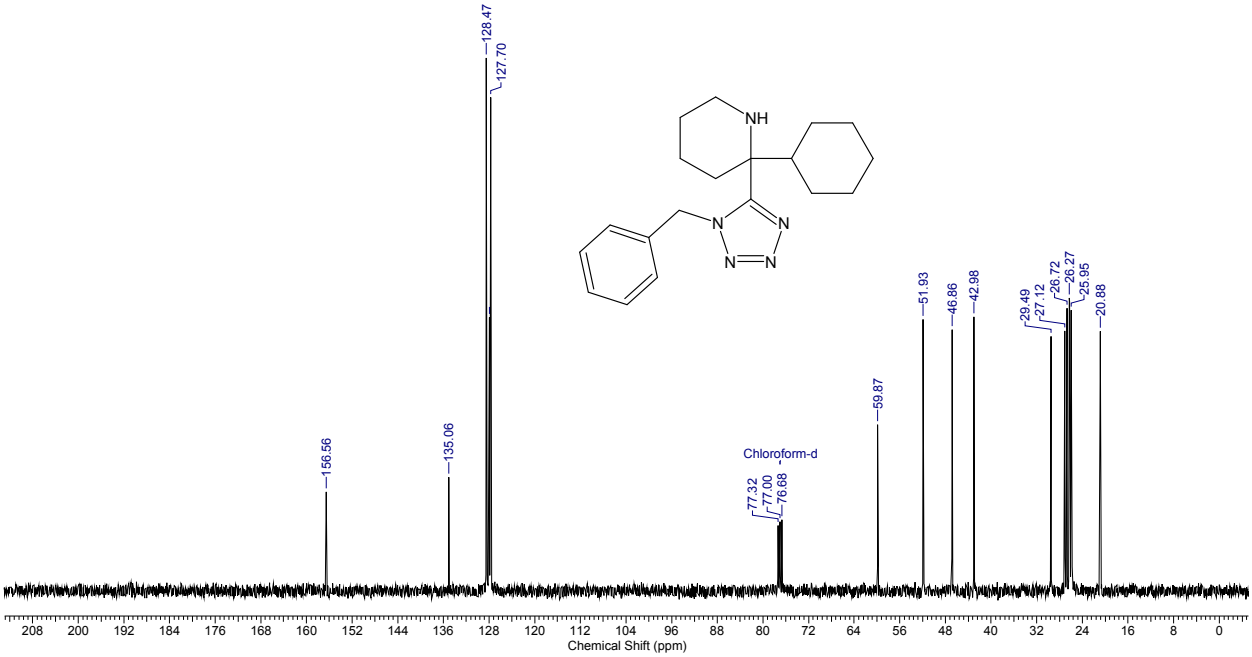
3j


7.23
7.25
7.27
7.22
7.27
7.28
7.29
7.20
6.00
5.96
6.04
5.92
3.62
3.05
3.03
3.03
3.01
3.01
2.76
2.72
2.62
2.49
2.46
2.25
2.26
2.24
2.28
2.18
2.16
2.15
1.97
acetone
1.86
1.84
1.83
1.55
1.53
1.35
1.34
1.28
1.26
1.25
5.42
2.00
1.13
0.98
0.99
1.01
5.11
3.23
6.10
Chemical Shift (ppm)

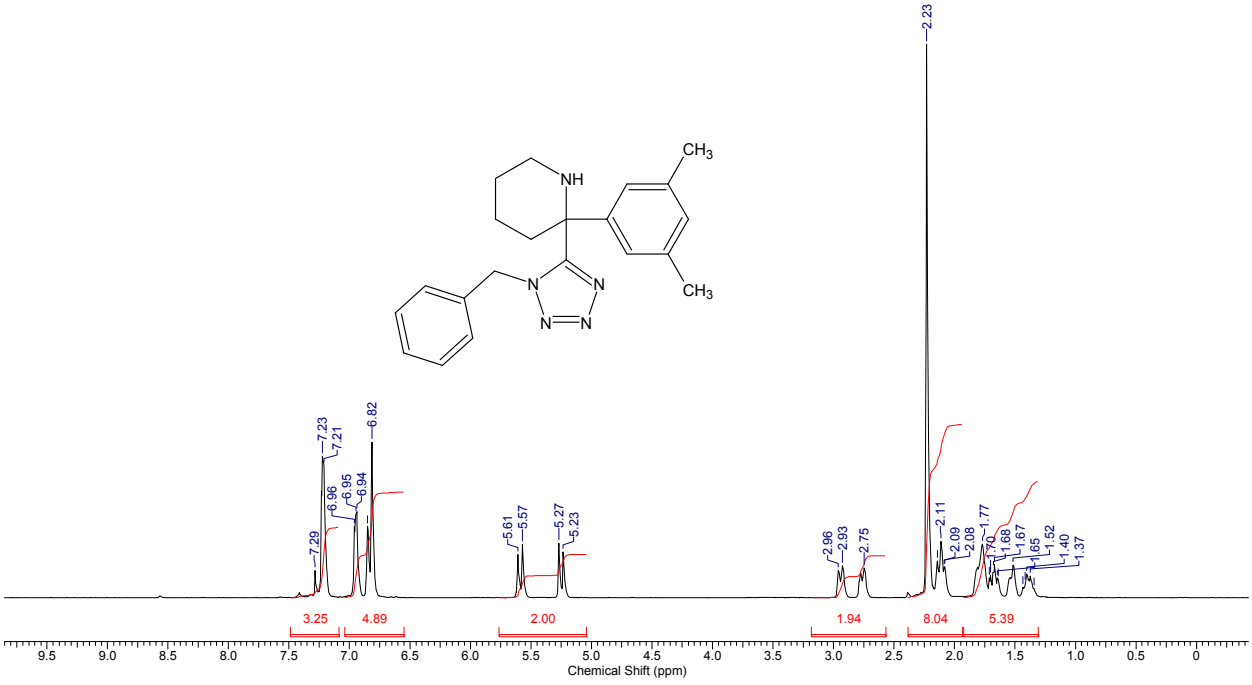

H
N
N
N
N
N
N
158.67
135.37
128.42
127.61
Chloroform-d
77.32
77.00
76.68
65.50
65.19
56.03
51.91
46.23
37.24
25.79
24.78
23.48
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

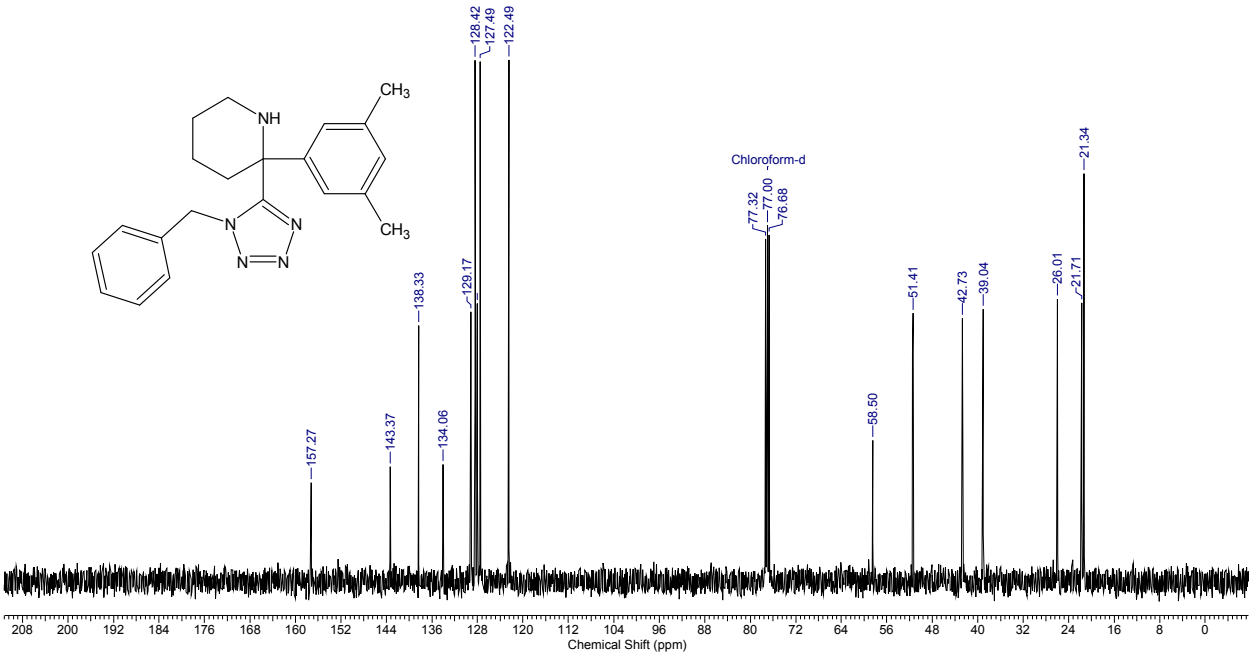
6a


NH
N
N
N
7.27
7.26
7.28
7.25
7.31
6.02
5.94
6.06
5.90
2.85
2.82
2.81
2.55
2.52
2.06
2.05
2.02
1.99
1.72
1.69
1.65
1.45
1.44
1.42
1.41
1.40
1.62
1.38
1.22
1.18
1.26
1.05
1.00
0.93
0.90
0.88
0.87
0.84
0.82
0.81
0.79
0.50
0.46
0.44
0.40
0.43
4.97
2.00
1.98
1.04
8.99
7.15
1.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0 -0.5
Chemical Shift (ppm)

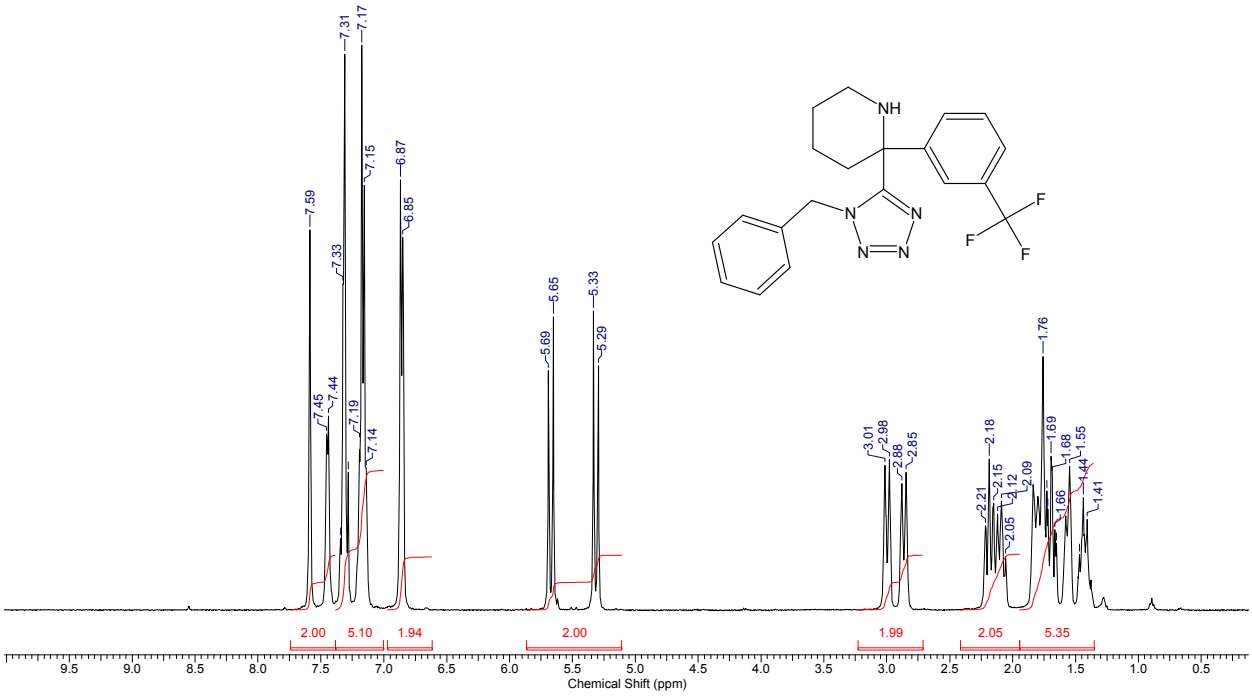

128.47
127.70
156.56
135.06
Chloroform-d
77.32
77.00
76.68
59.87
51.93
46.86
42.98
29.49
27.12
26.72
26.27
25.95
20.88
NH
N
N
N
N
Chemical Shift (ppm)

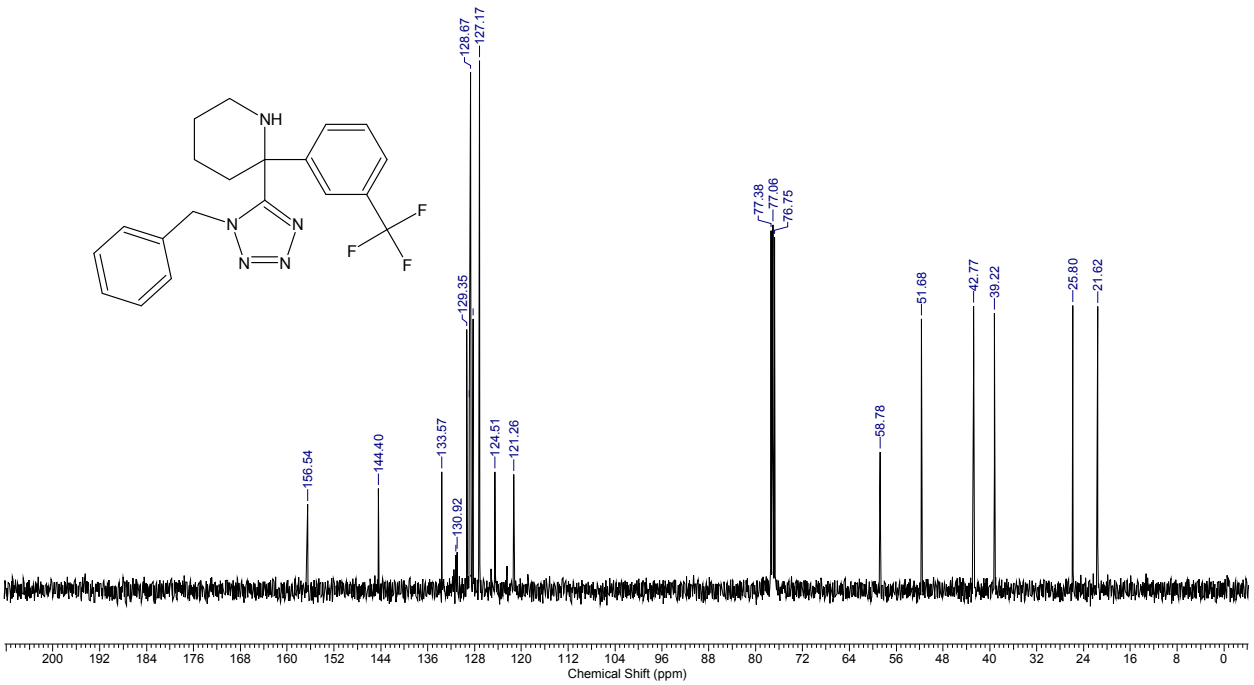
6b

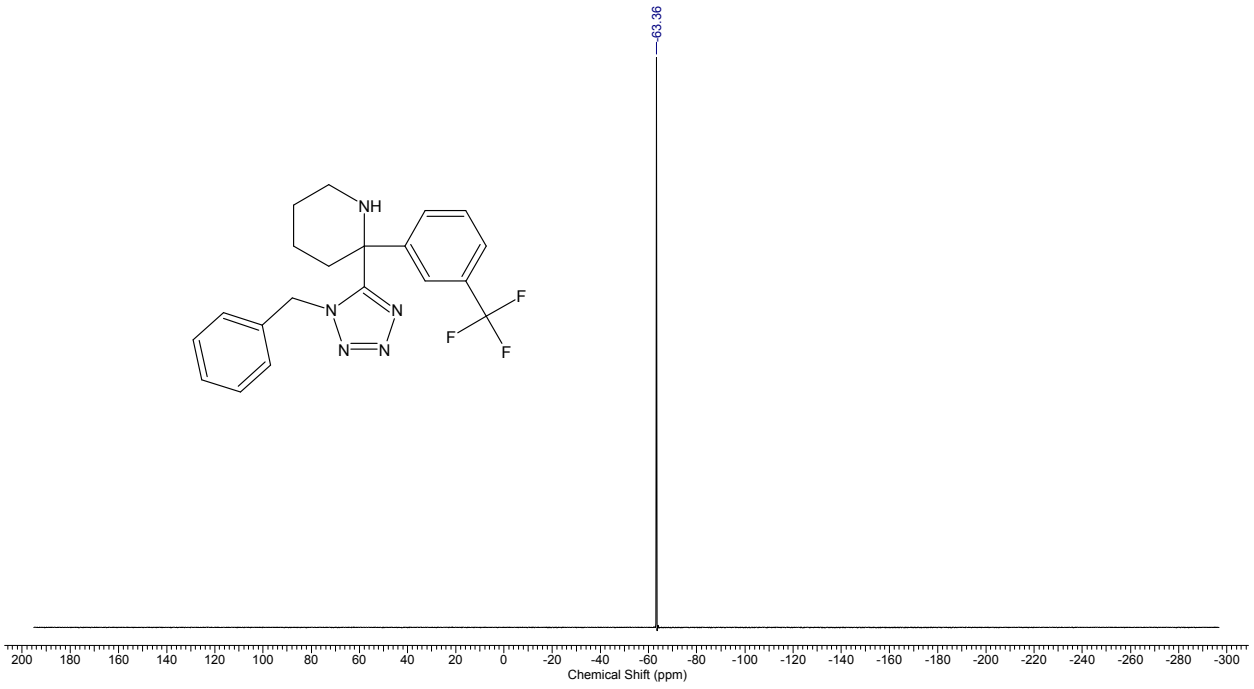

2.23
CH3
CH3
NH
N
N
N
N
7.23
7.21
6.82
6.96
6.95
6.94
7.29
5.61
5.57
5.27
5.23
2.96
2.93
2.75
2.11
2.09
2.08
1.77
1.70
1.68
1.67
1.65
1.52
1.40
1.37
3.25
4.89
2.00
1.94
8.04
5.39
Chemical Shift (ppm)


NH
CH3
CH3
N
N
N
N
128.42
127.49
122.49
138.33
129.17
Chloroform-d
77.32
77.00
76.68
21.34
157.27
143.37
134.06
58.50
51.41
42.73
39.04
26.01
21.71
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

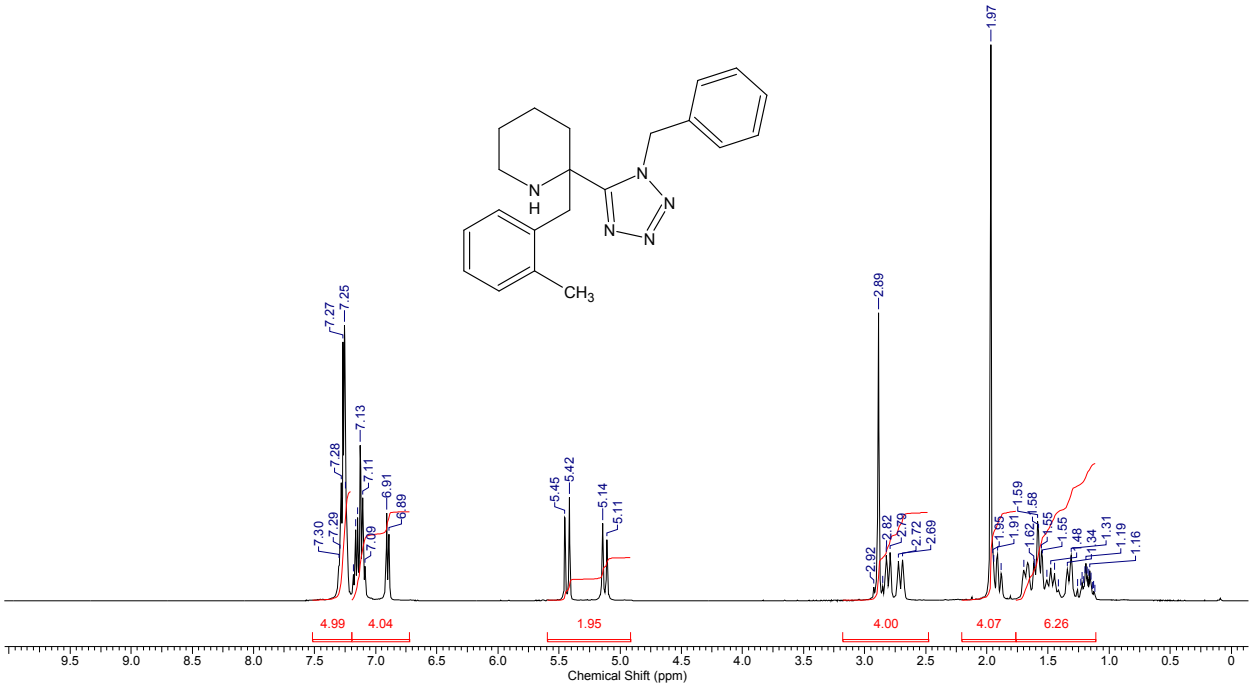
6c

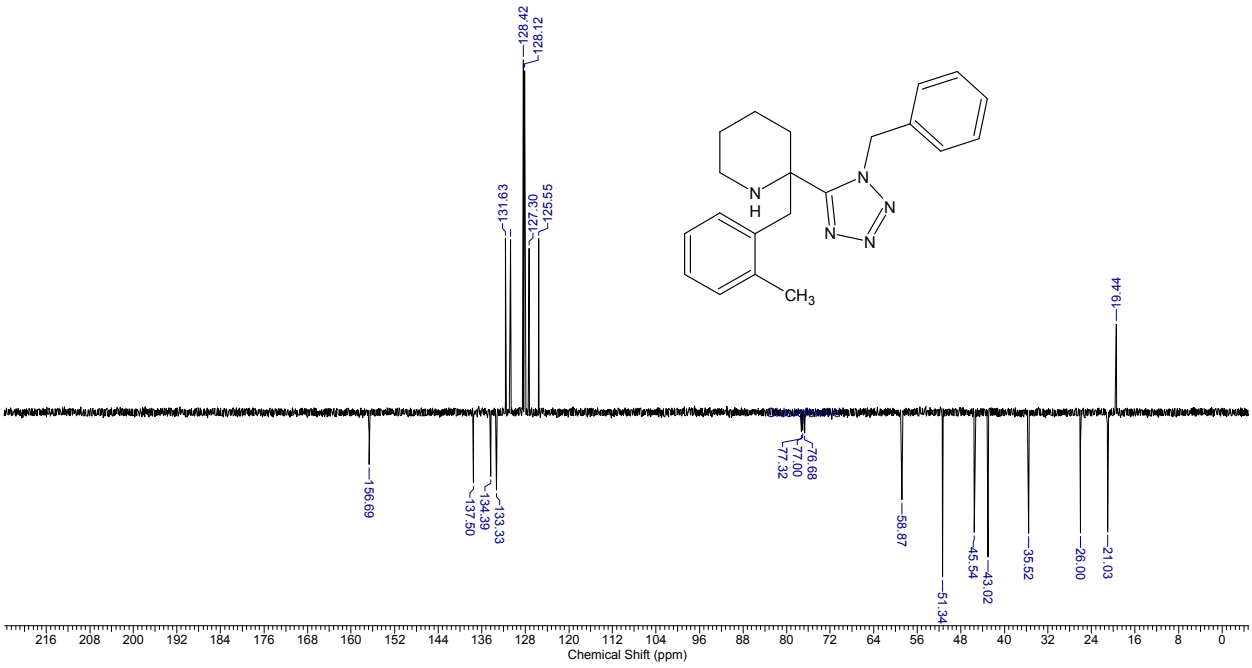

NH
N
N
N
N
F
F
F
7.31
7.17
7.15
6.87
6.85
7.59
7.33
7.45
7.44
7.19
7.14
5.65
5.33
5.69
5.29
1.76
3.01
2.98
2.88
2.85
2.18
1.69
1.68
1.55
2.21
2.15
2.12
2.09
1.66
2.05
1.44
1.41
2.00
5.10
1.94
2.00
1.99
2.05
5.35
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
Chemical Shift (ppm)


NH
N
N
N
N
F
F
F
128.67
127.17
129.35
77.38
77.06
76.75
51.68
42.77
39.22
25.80
21.62
58.78
156.54
144.40
133.57
130.92
124.51
121.26
200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

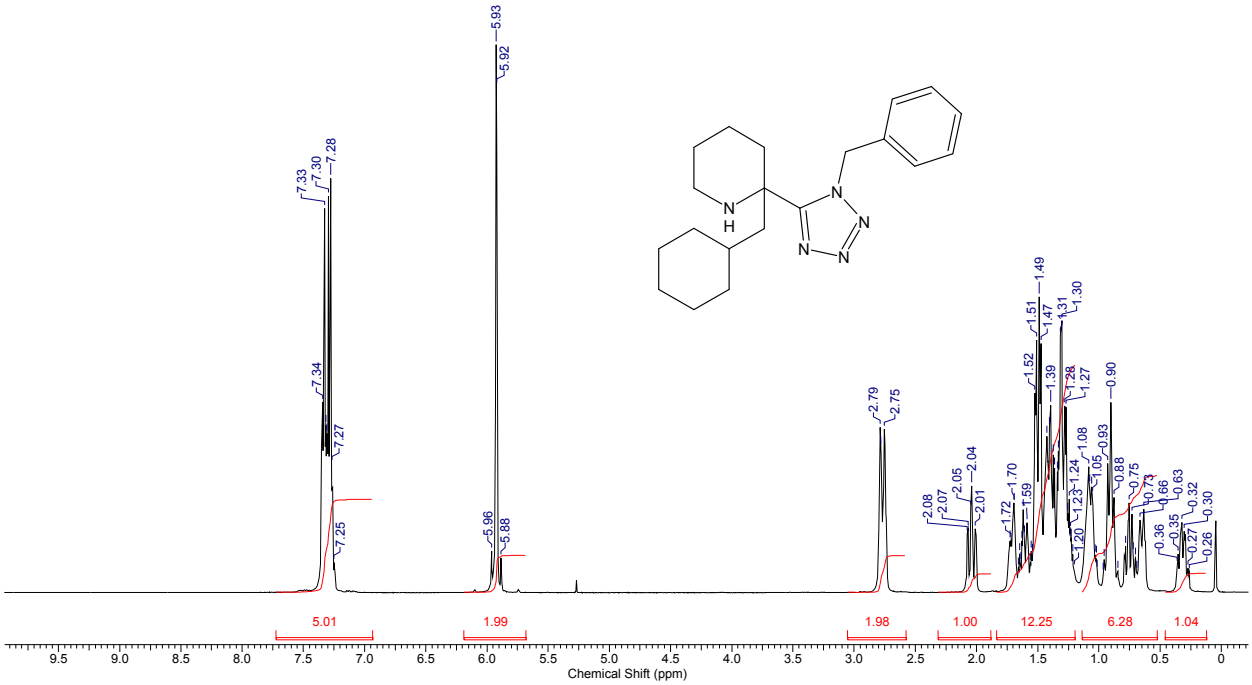

NH
N
N
N
N
F
F
F
63.36
200 180 160 140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 -300
Chemical Shift (ppm)

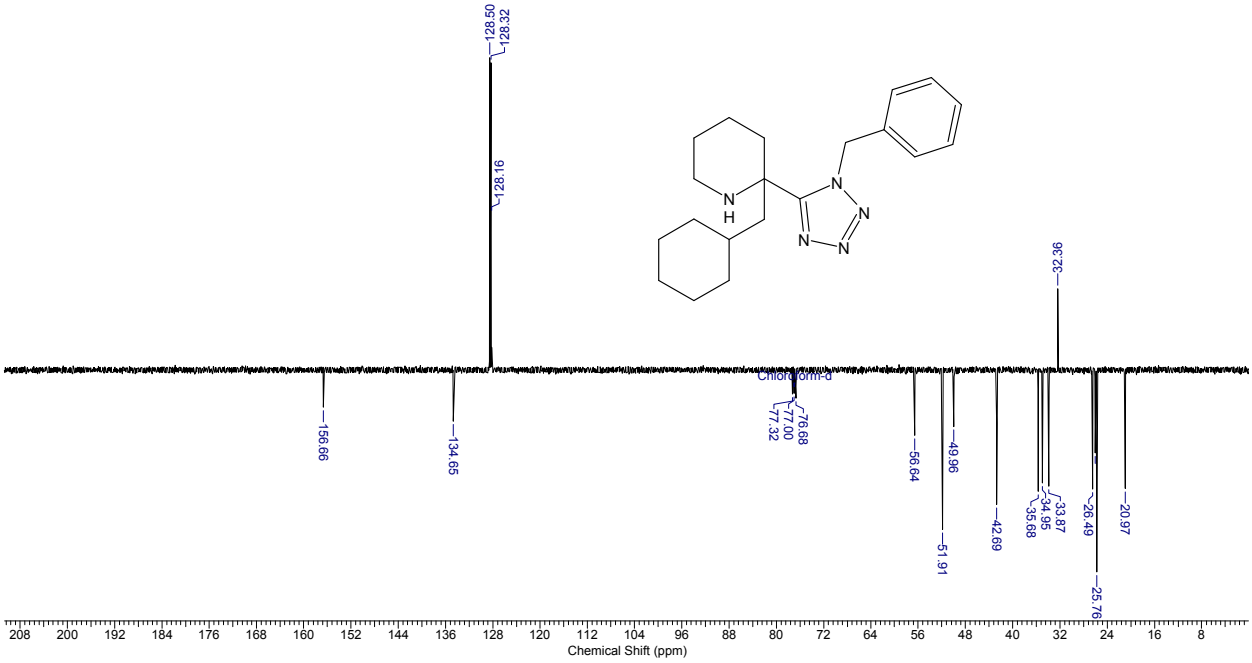
6d


7.27
7.25
7.28
7.13
7.11
7.30
7.29
7.09
6.91
6.89
5.45
5.42
5.14
5.11
2.89
2.92
2.82
2.79
2.72
2.69
1.97
1.95
1.91
1.62
1.59
1.58
1.55
1.55
1.48
1.34
1.31
1.19
1.16
4.99
4.04
1.95
4.00
4.07
6.26
Chemical Shift (ppm)

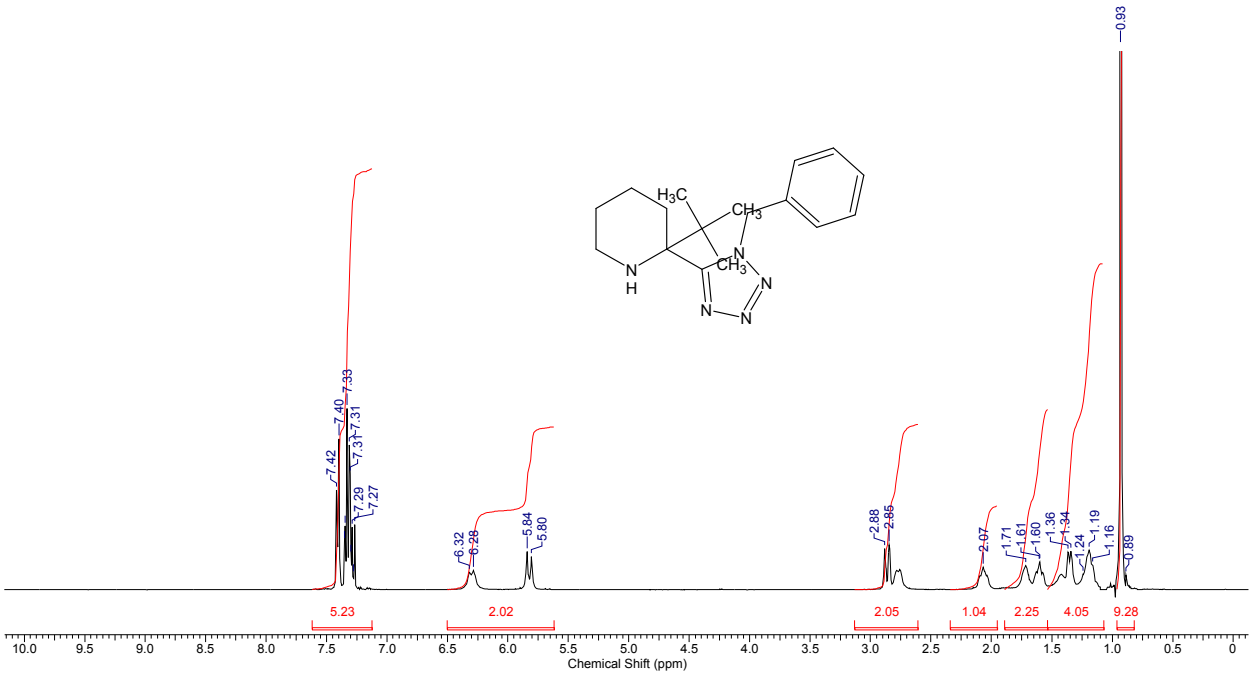

128.42
128.12
131.63
127.30
125.55
156.69
137.50
134.39
133.33
77.32
77.00
76.68
58.87
51.34
45.54
43.02
35.52
26.00
21.03
19.44
Chemical Shift (ppm)

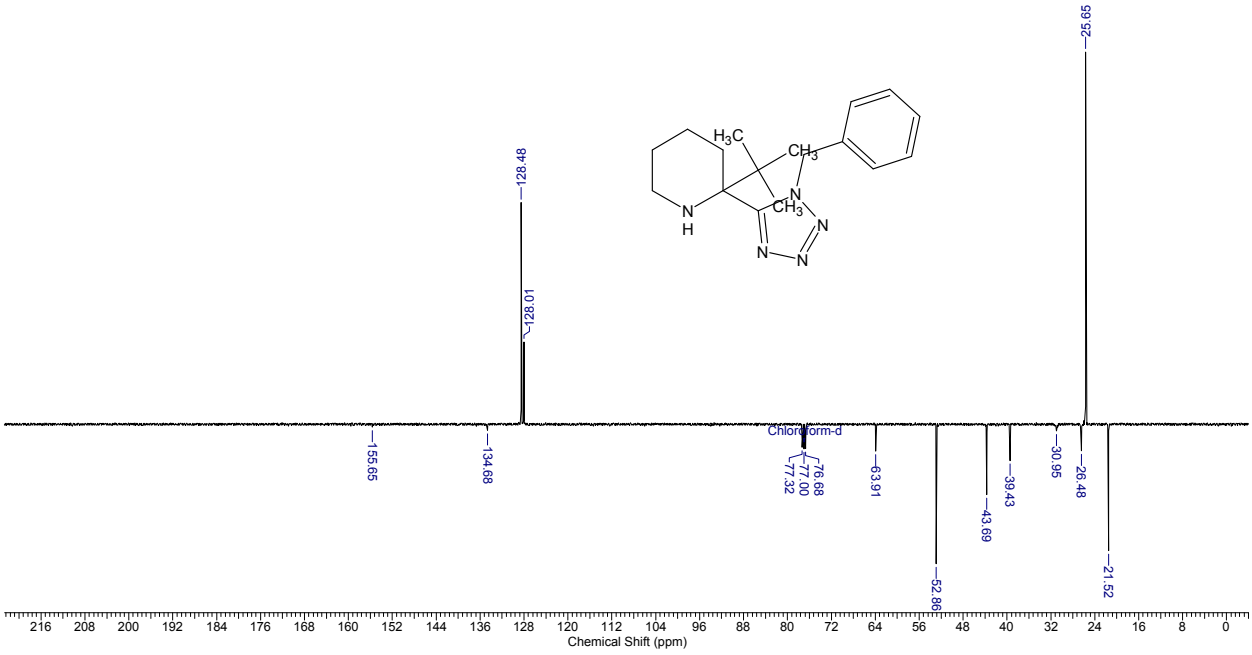
6e


7.33
7.30
7.28
7.34
7.27
7.25
5.93
5.92
5.96
5.88
2.79
2.75
2.08
2.07
2.05
2.04
2.01
1.72
1.70
1.59
1.52
1.51
1.49
1.47
1.39
1.31
1.30
1.28
1.27
1.24
1.23
1.20
1.08
1.05
0.93
0.90
0.88
0.75
0.73
0.68
0.63
0.36
0.35
0.32
0.30
0.27
0.26
N
H
N
N
N
5.01
1.99
1.98
1.00
12.25
6.28
1.04
Chemical Shift (ppm)

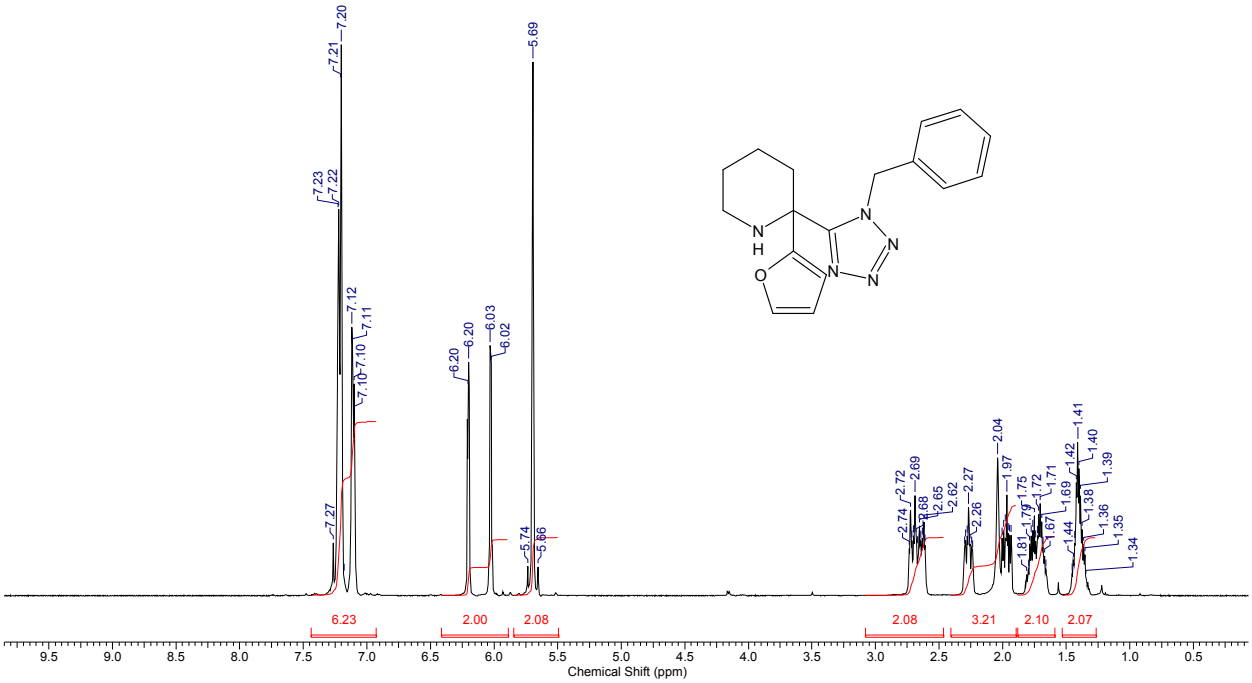

128.50
128.32
128.16
32.36
156.96
134.65
Chloroform-d
77.32
77.00
76.68
56.64
49.96
42.69
35.68
34.95
33.87
26.49
25.76
20.97
51.91
N
H
N
N
N
Chemical Shift (ppm)

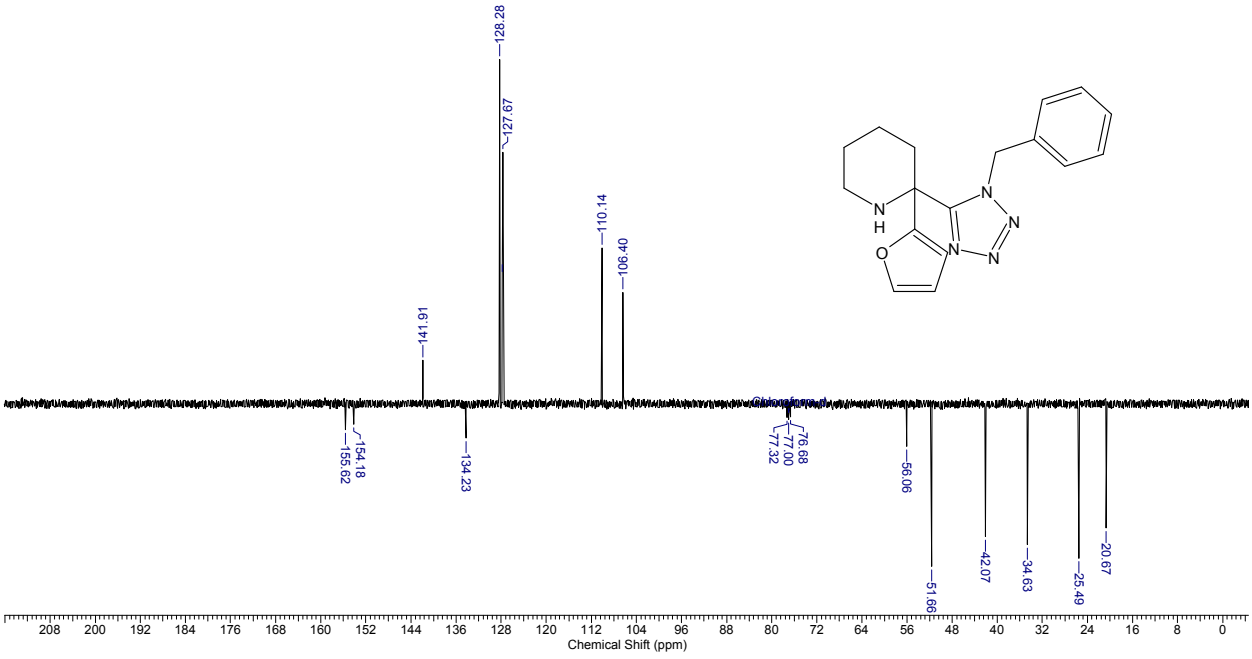
6f


0.93
7.42
7.40
7.33
7.31
7.31
7.29
7.27
6.32
6.28
5.84
5.80
2.88
2.86
2.07
1.71
1.61
1.60
1.36
1.34
1.24
1.19
1.16
0.89
5.23
2.02
2.05
1.04
2.25
4.05
9.28
Chemical Shift (ppm)

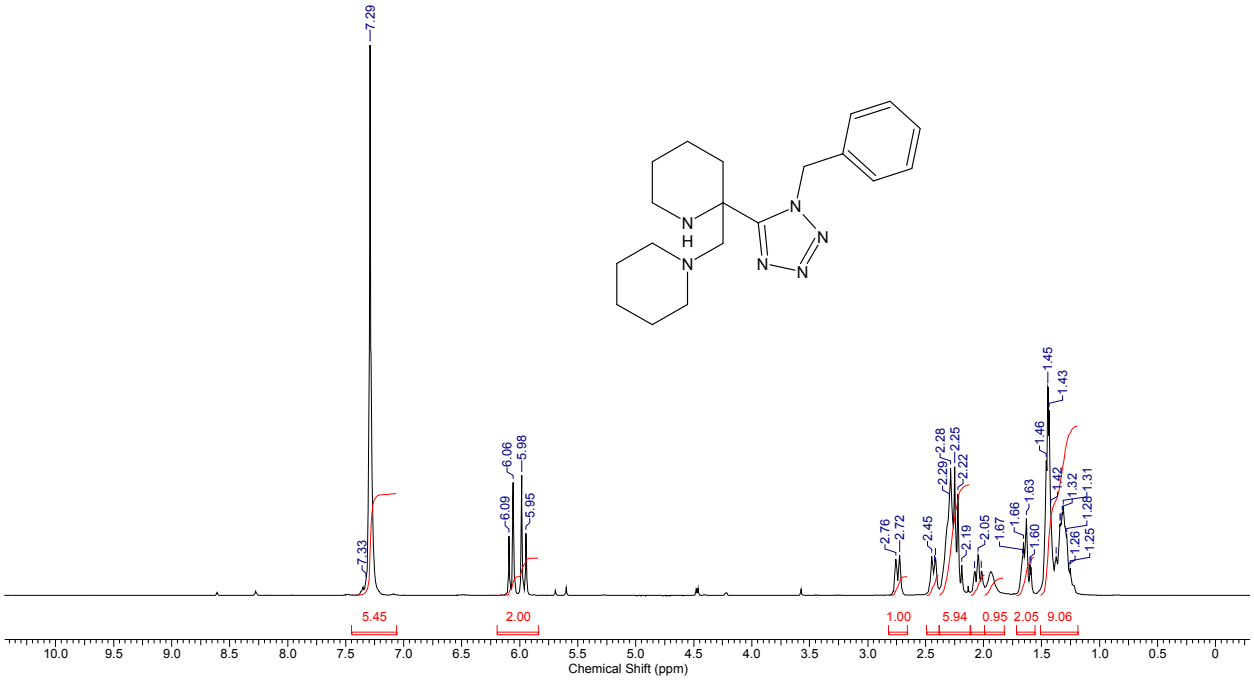

55.52
128.48
128.01
155.95
134.68
Chloroform-d
77.32
77.00
76.68
63.91
52.86
43.69
39.43
30.95
26.48
21.52
Chemical Shift (ppm)

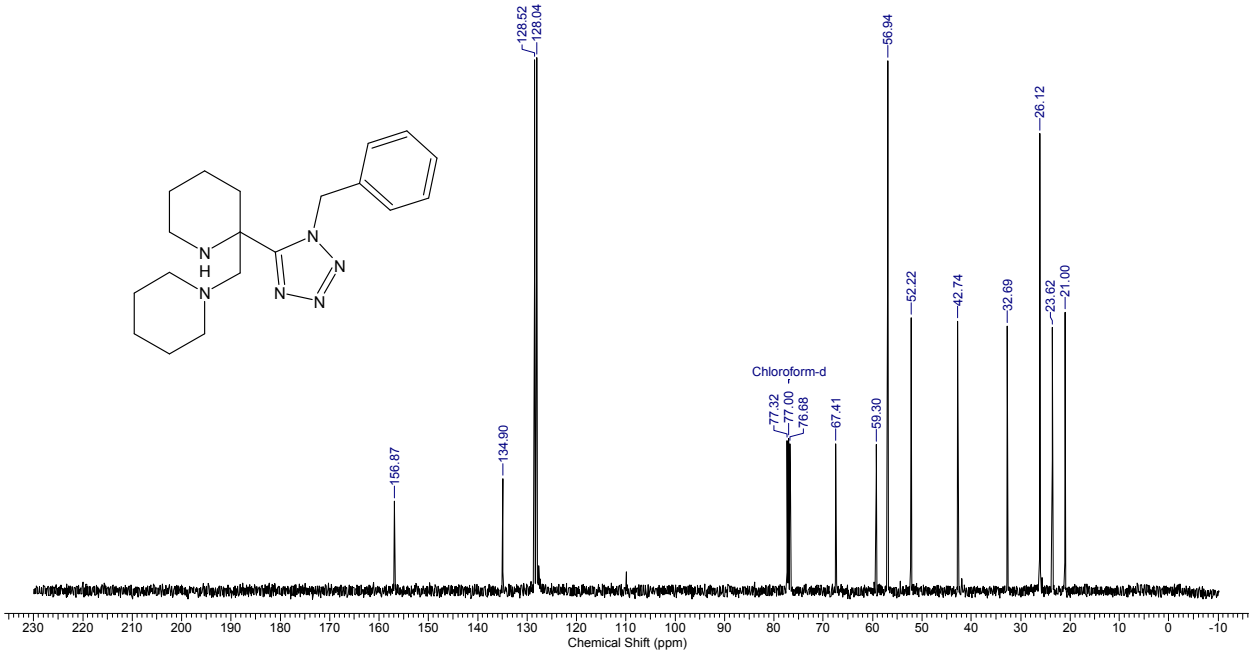
6g


7.27
7.23
7.22
7.21
7.20
7.12
7.11
7.10
7.10
6.20
6.20
6.03
6.02
5.74
5.66
5.69
2.74
2.72
2.69
2.68
2.65
2.62
2.27
2.26
2.04
1.97
1.81
1.79
1.75
1.72
1.71
1.67
1.69
1.44
1.42
1.41
1.40
1.39
1.38
1.36
1.35
1.34
6.23
2.00
2.08
2.08
3.21
2.10
2.07
Chemical Shift (ppm)

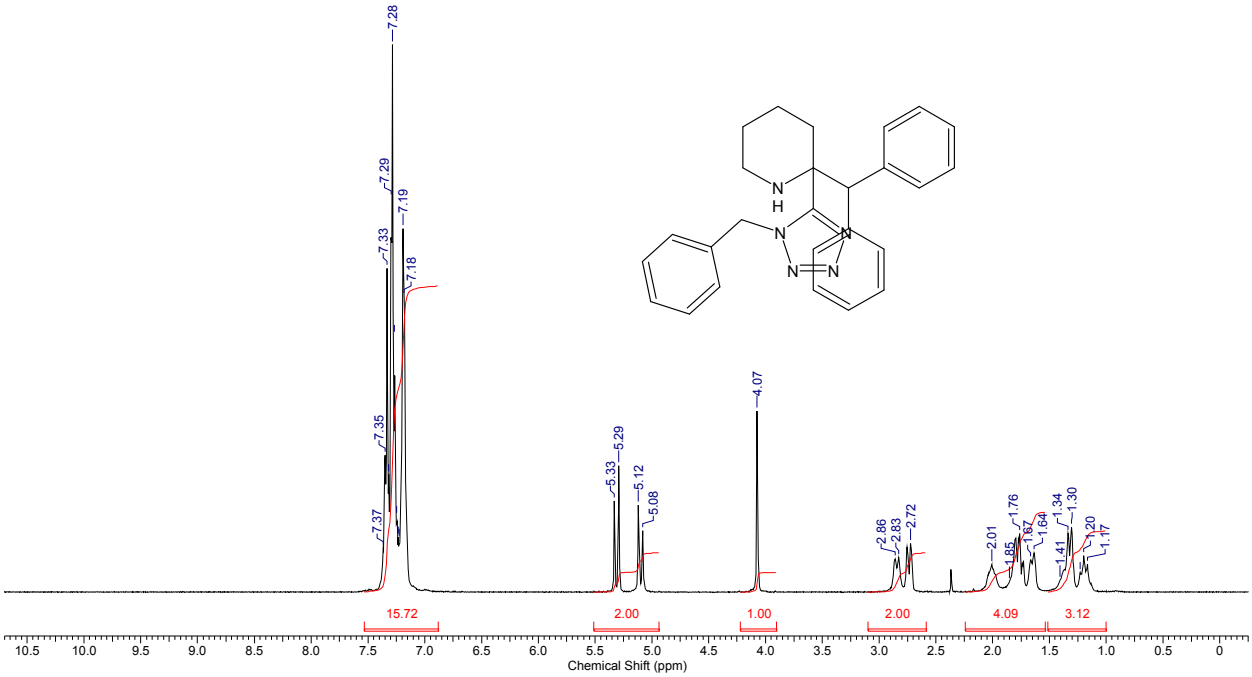

128.28
127.67
110.14
106.40
141.91
155.62
154.18
134.23
77.32
77.00
76.68
56.06
51.66
42.07
34.63
25.49
20.67
Chemical Shift (ppm)

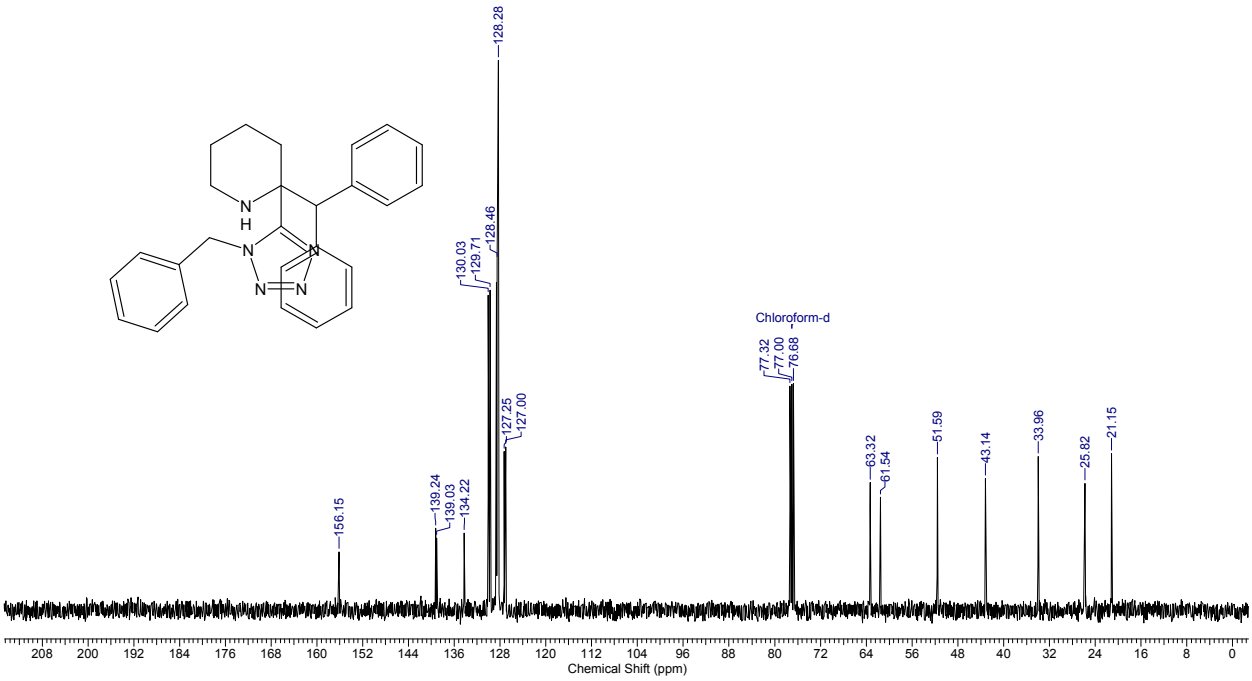
6h


7.29
7.33
6.09
6.06
5.98
5.95
2.76
2.72
2.45
2.29
2.28
2.25
2.22
2.19
2.05
1.67
1.66
1.63
1.60
1.46
1.45
1.43
1.42
1.32
1.31
1.28
1.26
1.25
5.45
2.00
1.00
5.94
0.95
2.05
9.06
Chemical Shift (ppm)

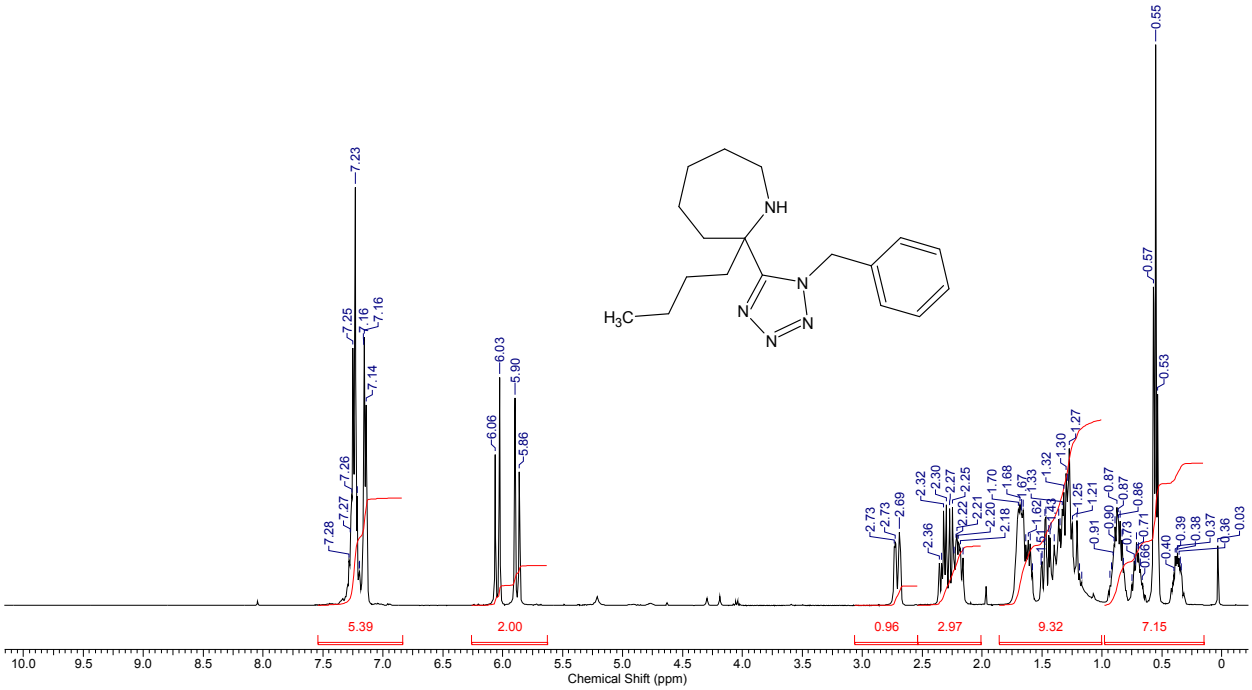

128.52
128.04
56.94
26.12
52.22
42.74
32.69
23.62
21.00
Chloroform-d
77.32
77.00
76.68
67.41
59.30
156.87
134.90
Chemical Shift (ppm)

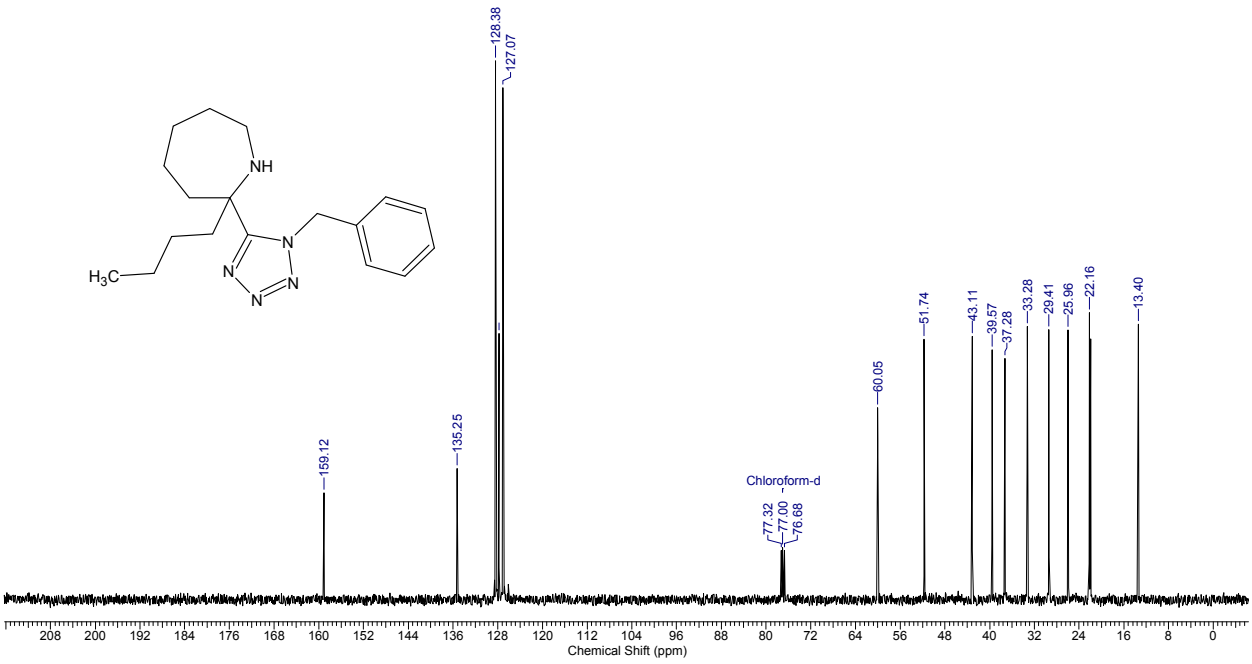
6i


7.28
7.29
7.19
7.33
7.18
7.35
7.37
5.33
5.29
5.12
5.08
4.07
2.86
2.83
2.72
2.01
1.95
1.76
1.67
1.64
1.41
1.34
1.30
1.20
1.17
15.72
2.00
1.00
2.00
4.09
3.12
Chemical Shift (ppm)

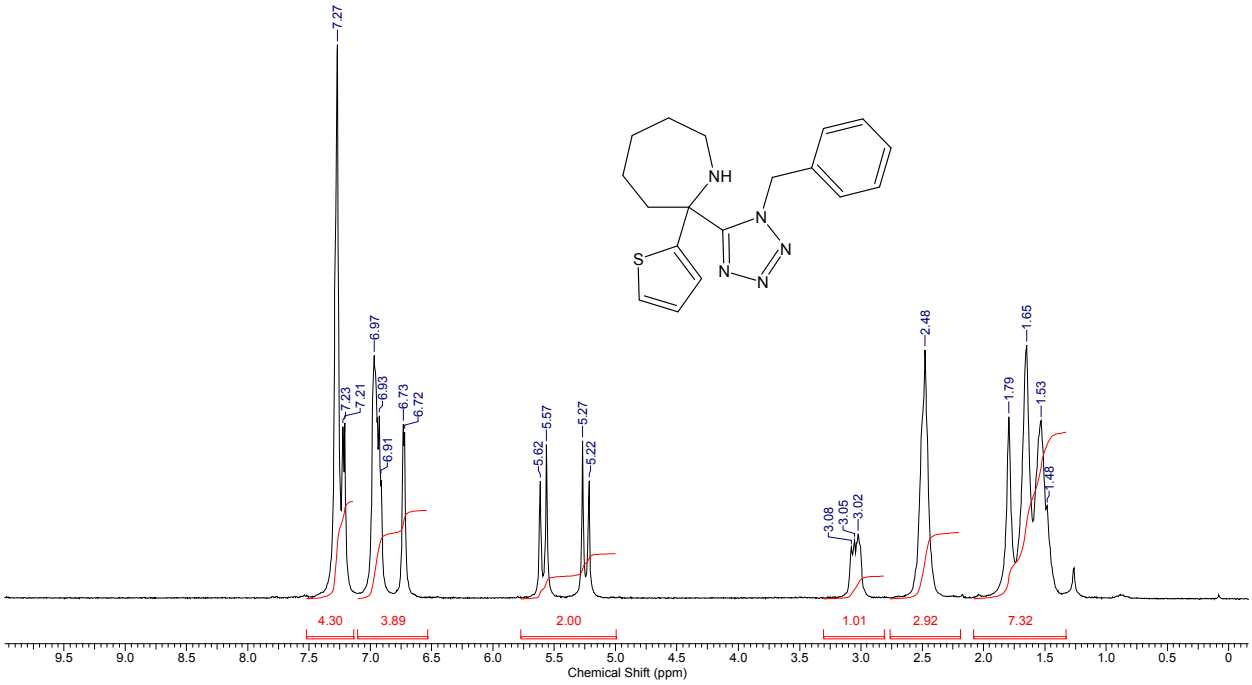

128.28
130.03
129.71
128.46
127.25
127.00
Chloroform-d
77.32
77.00
76.68
156.15
139.24
139.03
134.22
63.32
61.54
51.59
43.14
33.96
25.82
21.15
Chemical Shift (ppm)

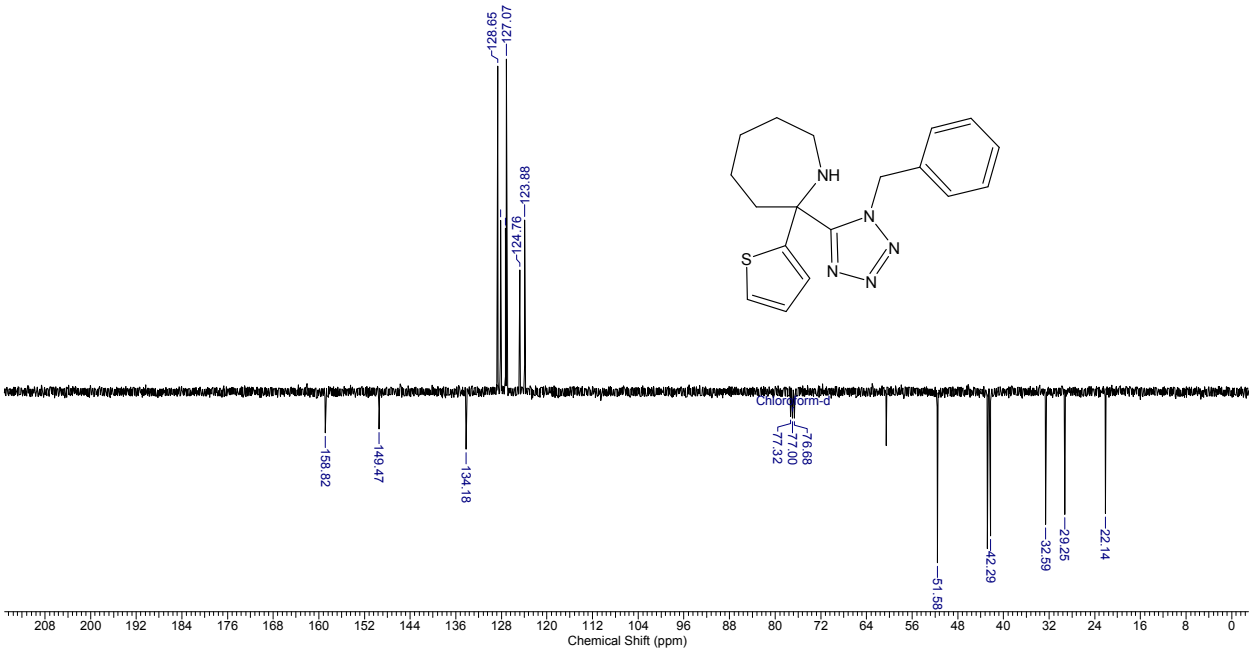
8a


NH
N
N
N
H3C
7.23
7.25
7.26
7.16
7.14
7.28
7.27
6.03
5.90
6.06
5.86
0.55
0.57
0.53
1.27
1.30
1.32
1.33
1.67
1.68
1.70
2.18
2.20
2.21
2.22
2.25
2.27
2.30
2.32
2.36
2.69
2.73
2.73
0.87
0.87
0.86
0.71
0.40
0.39
0.38
0.37
0.36
0.03
5.39
2.00
0.96
2.97
9.32
7.15
Chemical Shift (ppm)

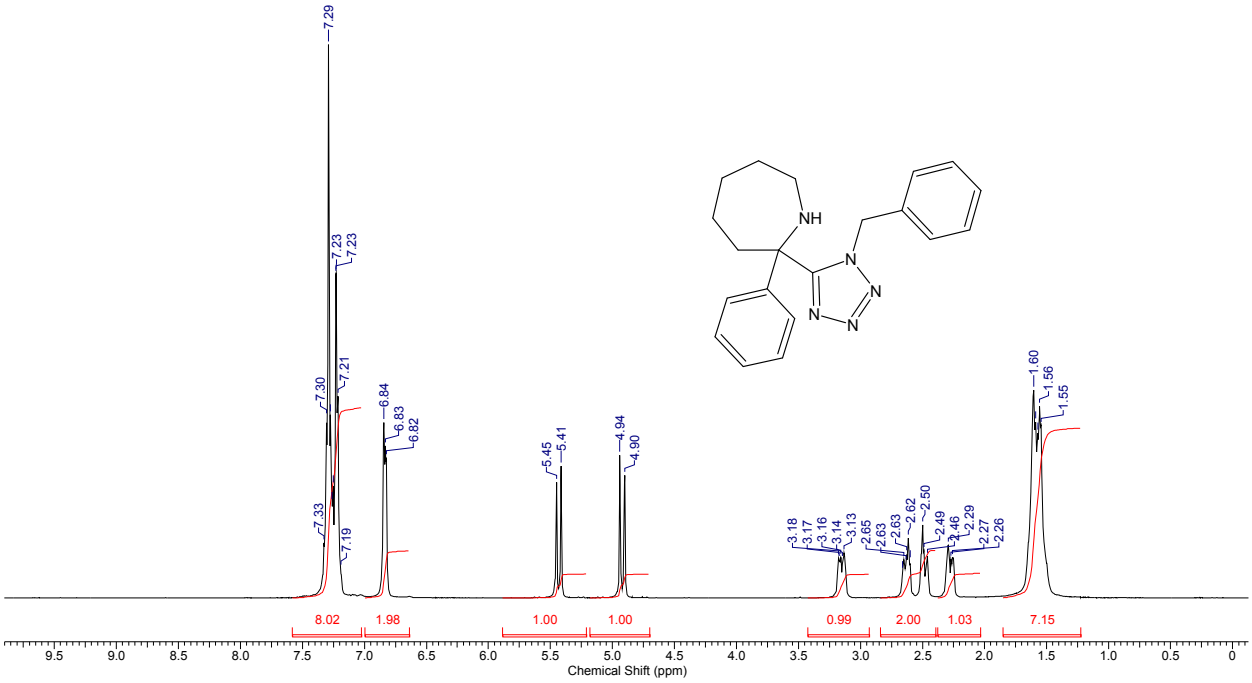

NH
N
N
N
H3C
128.38
127.07
159.12
135.25
Chloroform-d
77.32
77.00
76.68
60.05
51.74
43.11
39.57
37.28
33.28
29.41
25.96
22.16
13.40
Chemical Shift (ppm)

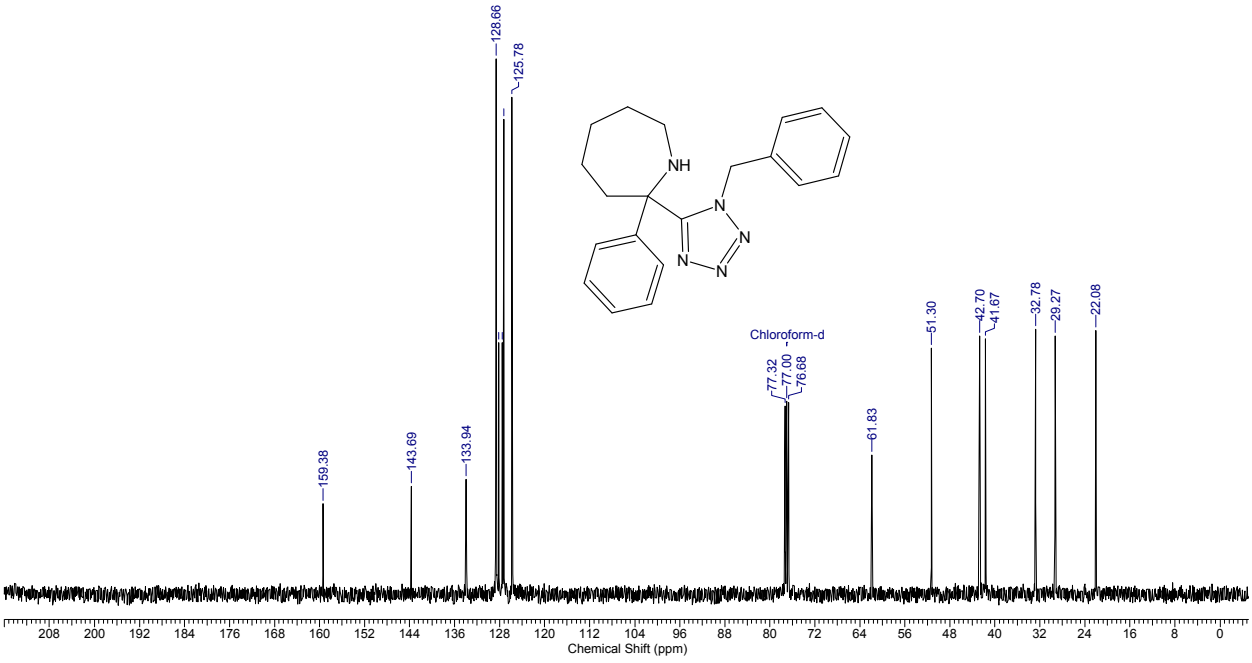
8b


7.27
7.23
7.21
6.97
6.93
6.91
6.73
6.72
5.62
5.57
5.27
5.22
3.08
3.05
3.02
2.48
1.79
1.65
1.53
1.48
NH
S
N
N
N
N
4.30
3.89
2.00
1.01
2.92
7.32
Chemical Shift (ppm)

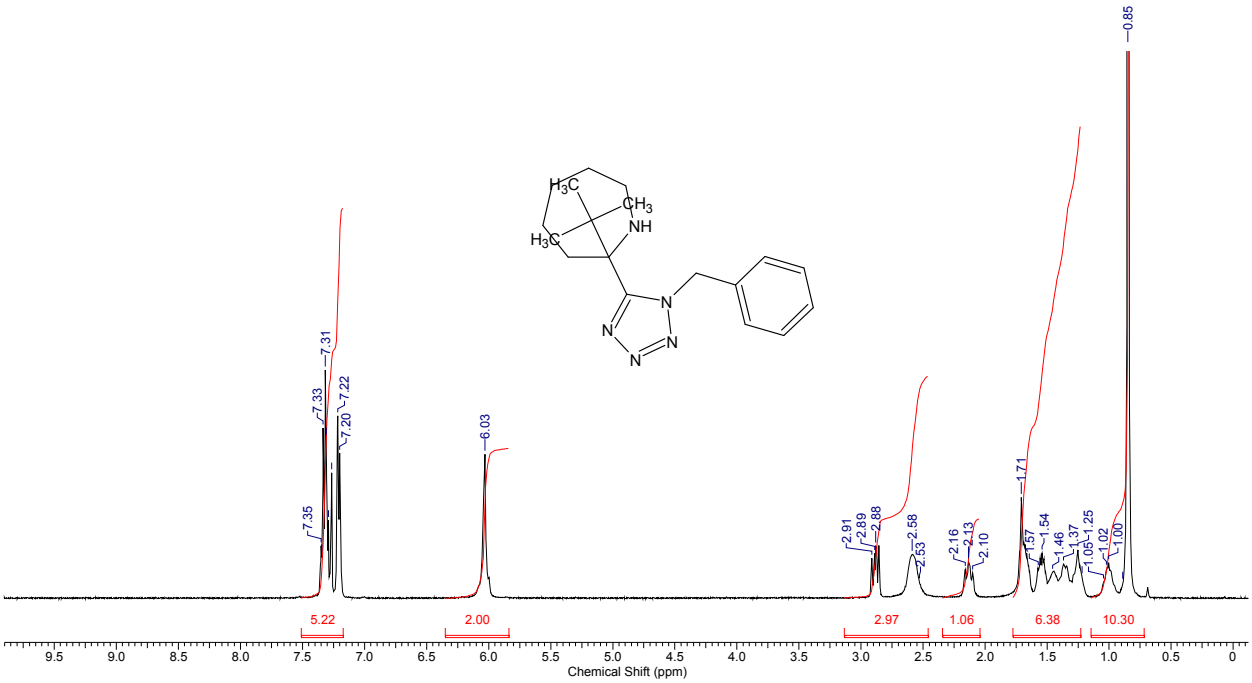

128.65
127.07
123.88
124.76
158.82
149.47
134.18
Chloroform-d
77.32
77.00
76.68
51.58
42.29
32.59
29.25
22.14
NH
S
N
N
N
N
Chemical Shift (ppm)

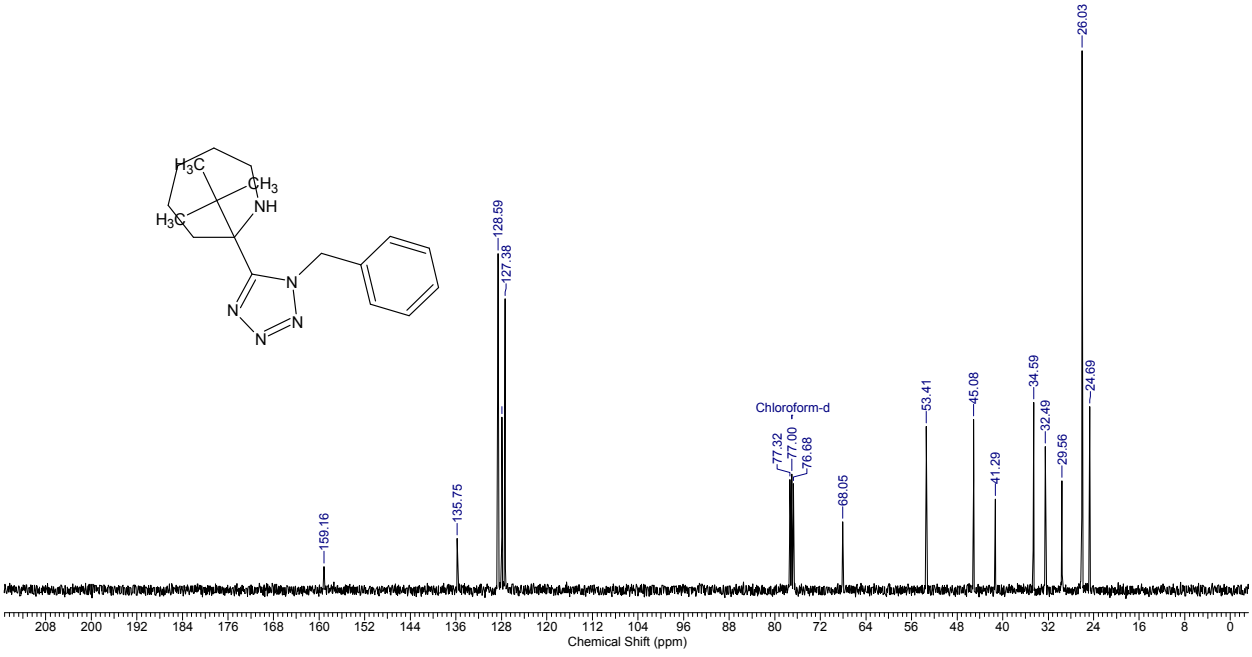
8c


7.29
7.23
7.23
7.30
7.21
6.84
6.83
6.82
7.33
7.19
5.45
5.41
4.94
4.90
3.18
3.17
3.16
3.14
3.13
2.65
2.63
2.62
2.50
2.49
2.46
2.29
2.27
2.26
1.60
1.56
1.55
NH
N
N
N
8.02
1.98
1.00
1.00
0.99
2.00
1.03
7.15
Chemical Shift (ppm)

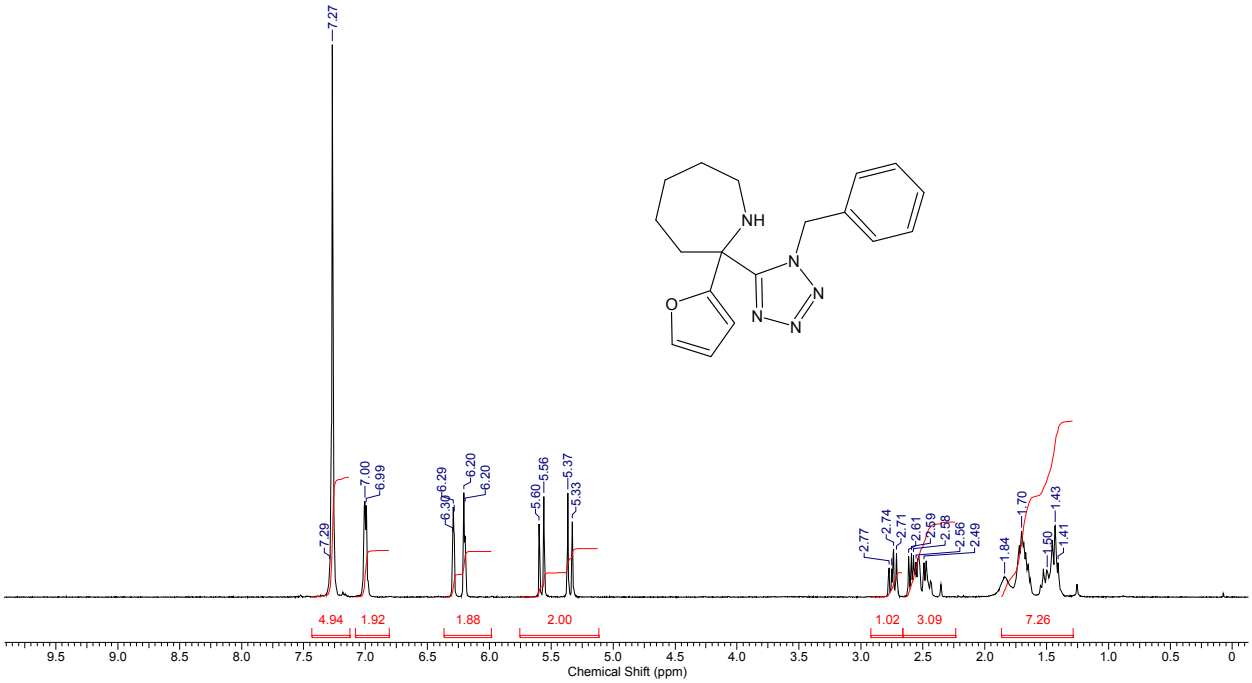

128.66
125.78
159.38
143.69
133.94
Chloroform-d
77.32
77.00
76.68
61.83
51.30
42.70
41.67
32.78
29.27
22.08
NH
N
N
N
Chemical Shift (ppm)

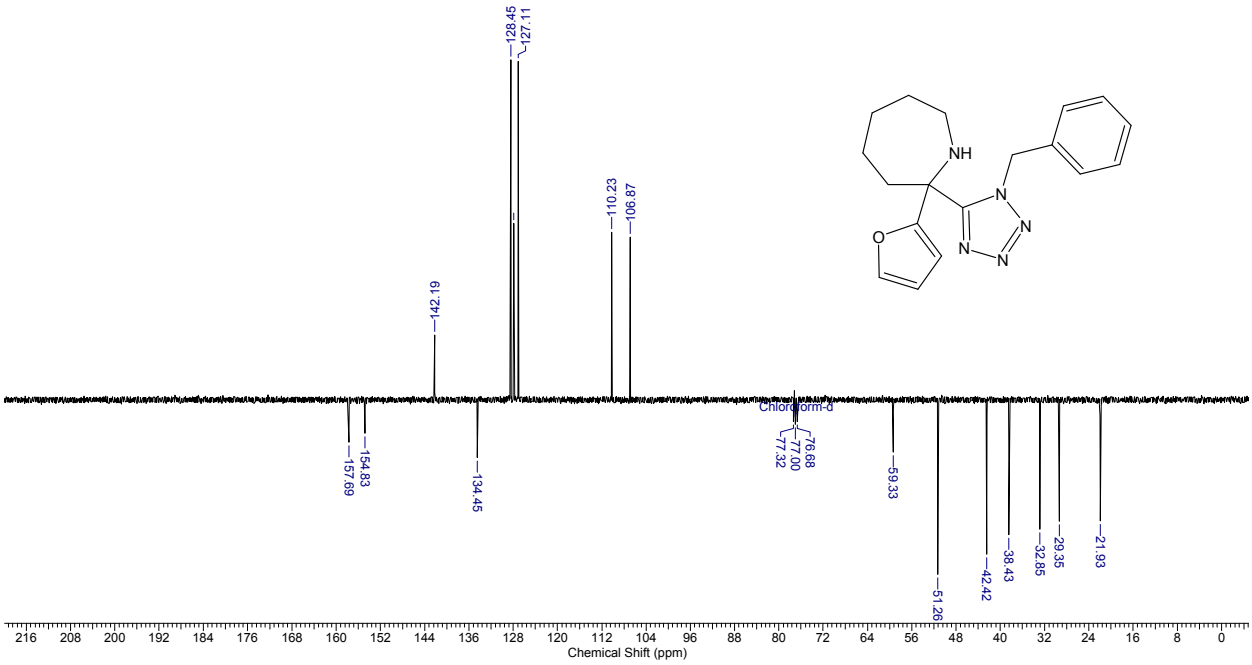
8d


0.85
7.35
7.33
7.31
7.22
7.20
6.03
2.91
2.89
2.88
2.58
2.53
2.16
2.13
2.10
1.71
1.57
1.54
1.46
1.37
1.25
1.05
1.02
1.00
5.22
2.00
2.97
1.06
6.38
10.30
Chemical Shift (ppm)

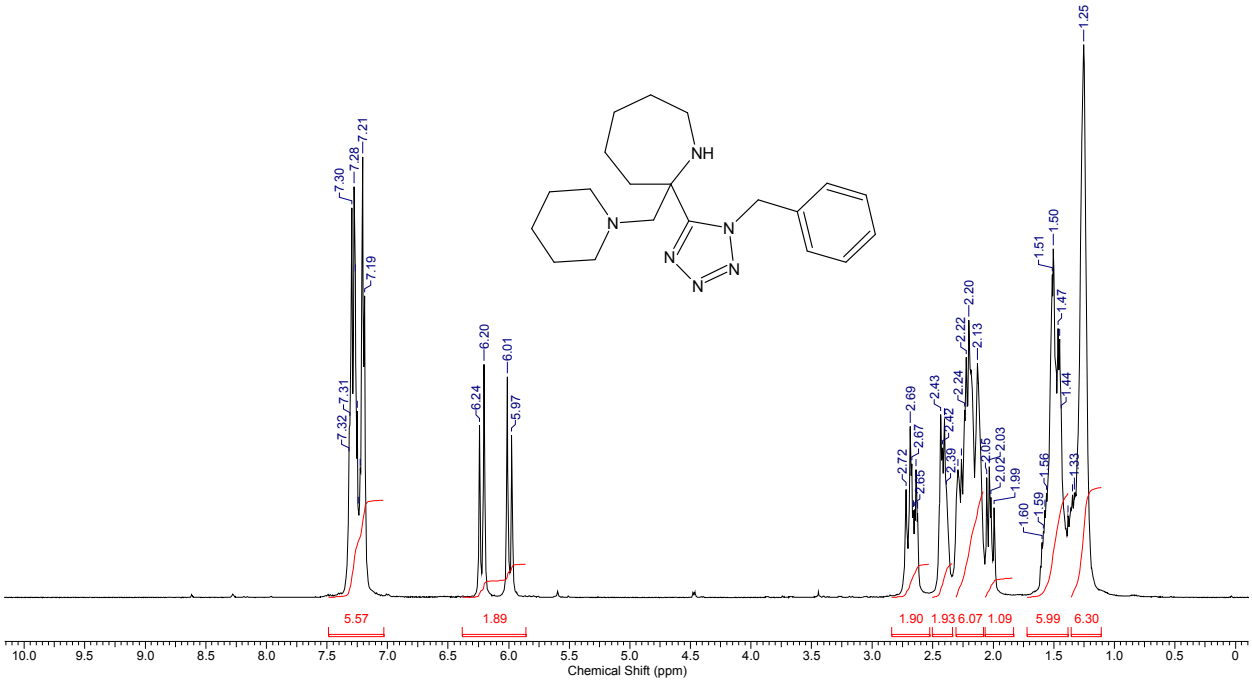

26.03
128.59
127.38
Chloroform-d
77.32
77.00
76.68
53.41
45.08
34.59
32.49
29.56
24.69
41.29
68.05
159.16
135.75
Chemical Shift (ppm)

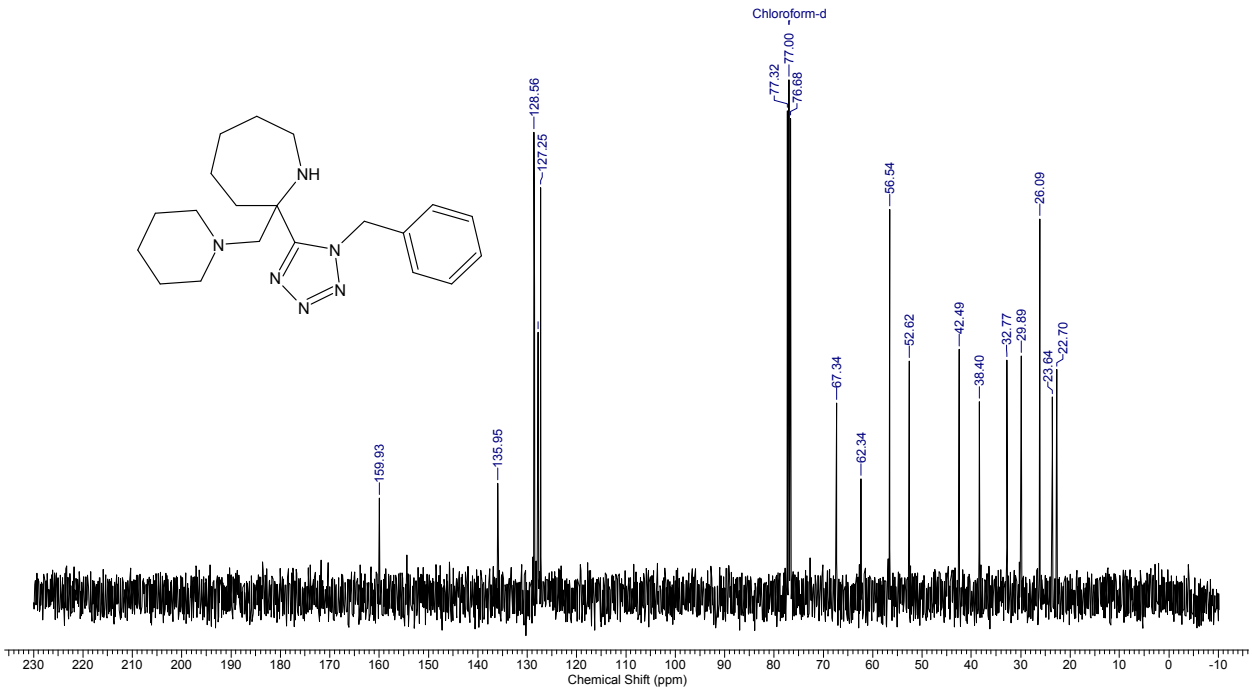
8e


7.27
7.29
7.00
6.99
6.39
6.29
6.20
6.20
5.60
5.56
5.37
5.33
2.77
2.74
2.71
2.61
2.59
2.58
2.56
2.49
1.84
1.70
1.50
1.43
1.41
4.94
1.92
1.88
2.00
1.02
3.09
7.26
NH
O
N
N
N
Chemical Shift (ppm)

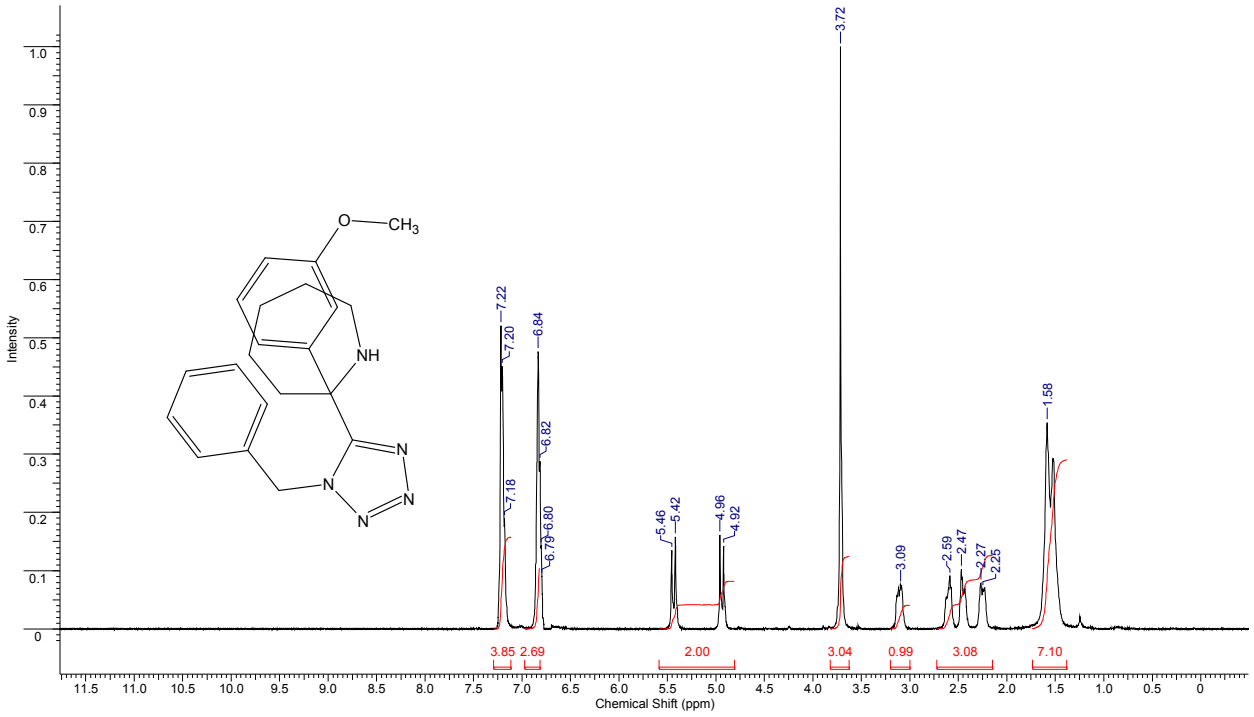

128.45
127.11
142.19
110.23
106.87
157.69
154.83
134.45
Chloroform-d
77.32
77.00
76.68
59.33
51.26
42.42
38.43
32.85
29.35
21.93
NH
O
N
N
N
Chemical Shift (ppm)

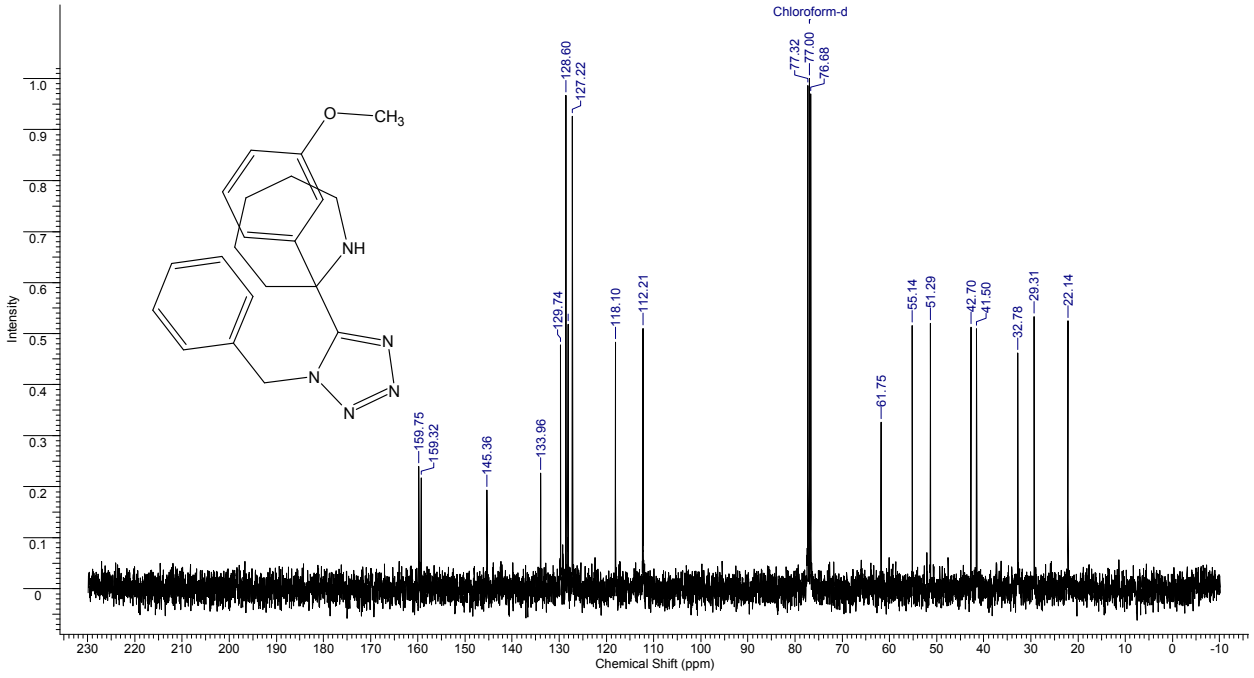
8f


NH
N
N
N
N
7.30
7.28
7.21
7.19
7.32
7.31
6.20
6.24
6.01
5.97
2.69
2.72
2.67
2.65
2.43
2.42
2.39
2.24
2.22
2.20
2.13
2.05
2.03
2.02
1.99
1.60
1.59
1.56
1.51
1.50
1.47
1.44
1.33
1.25
5.57
1.89
1.90
1.93
6.07
1.09
5.99
6.30
Chemical Shift (ppm)

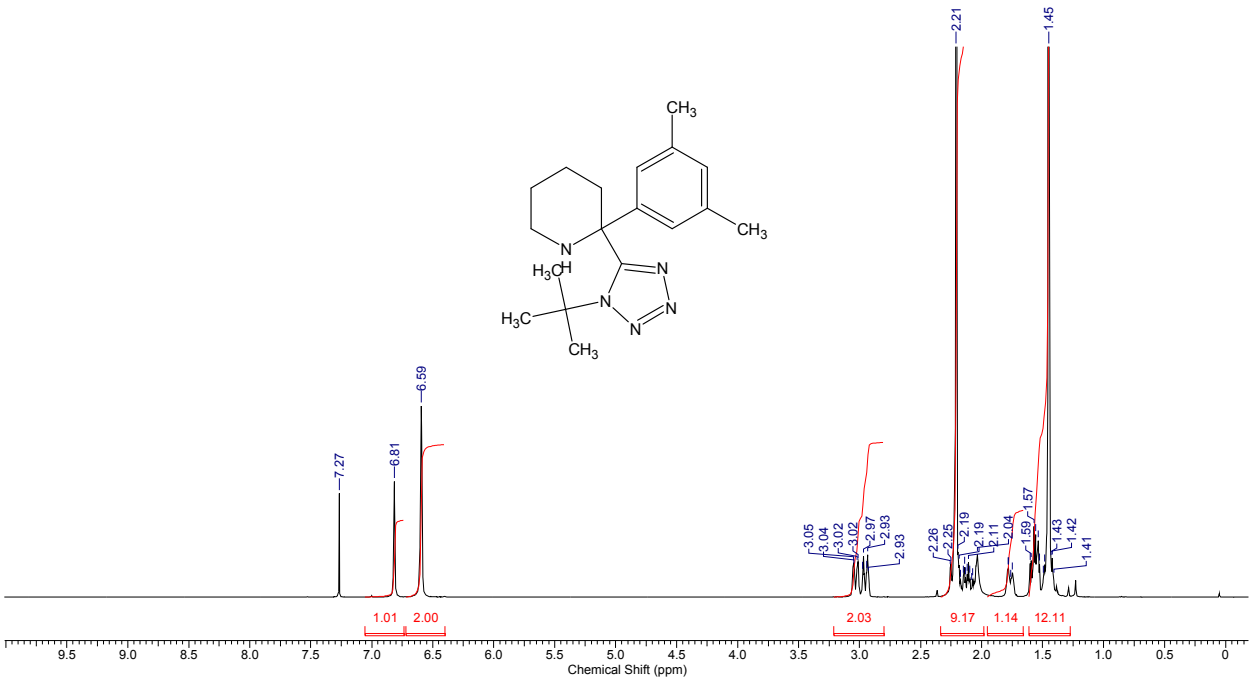

NH
N
N
N
N
Chloroform-d
77.32
77.00
76.68
128.56
127.25
159.93
135.95
67.34
62.34
56.54
52.62
42.49
38.40
32.77
29.89
26.09
23.64
22.70
Chemical Shift (ppm)

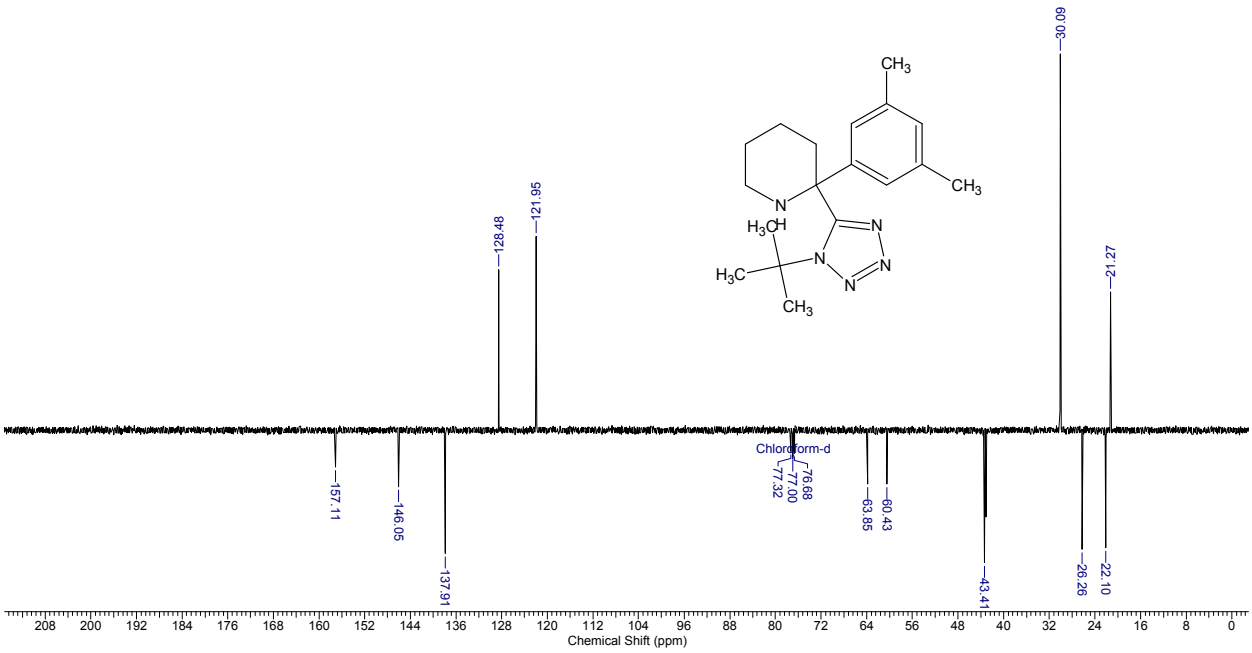
8g


O
CH3
NH
N
N
N
Intensity
Chemical Shift (ppm)
7.22
7.20
7.18
6.84
6.82
6.80
6.79
5.46
5.42
4.96
4.92
3.72
3.09
2.59
2.47
2.27
2.25
1.58
3.85
2.69
2.00
3.04
0.99
3.08
7.10

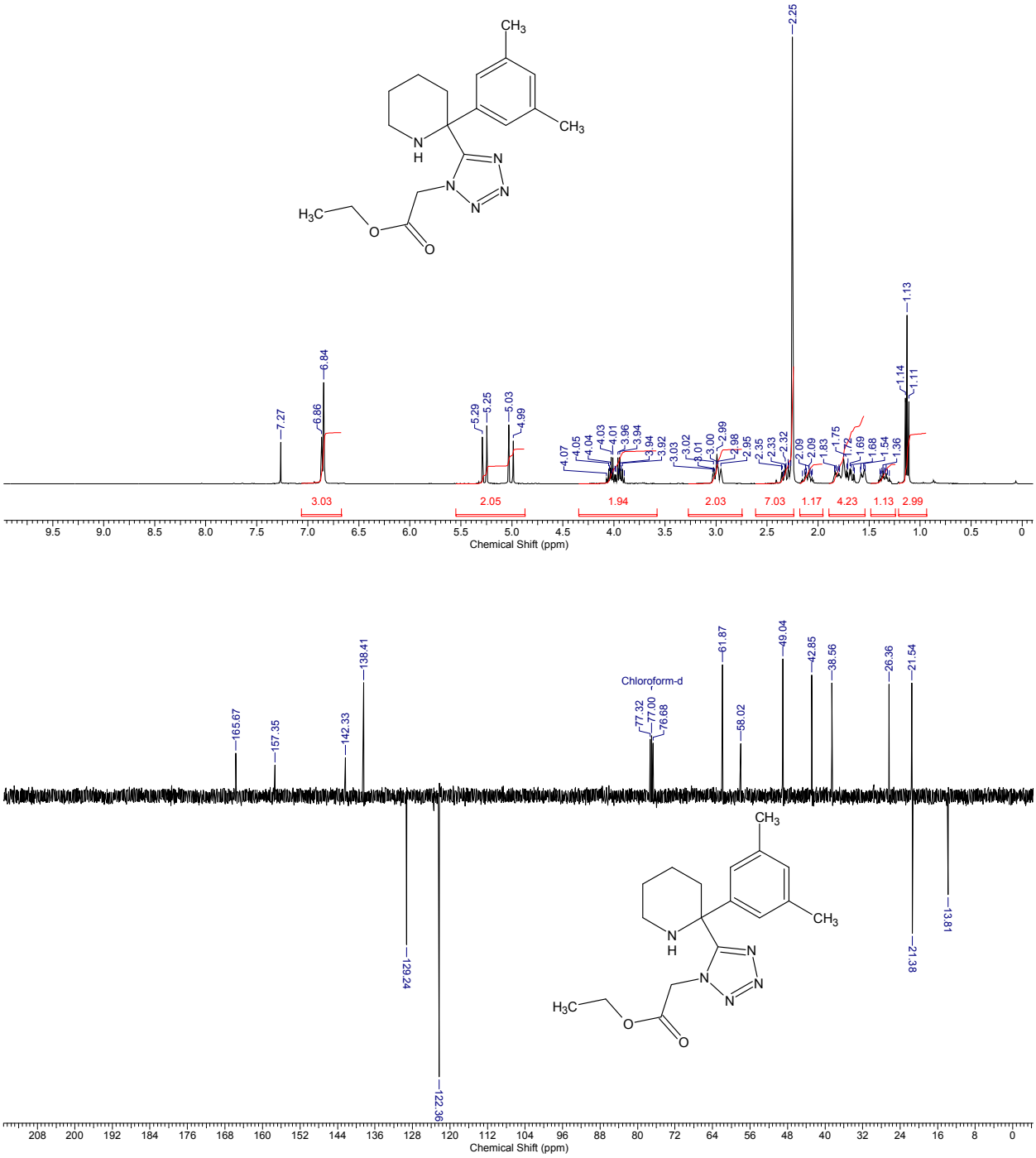

O
CH3
NH
N
N
N
Intensity
Chemical Shift (ppm)
Chloroform-d
77.32
77.00
76.68
128.60
127.22
129.74
118.10
112.21
159.75
159.32
145.36
133.96
61.75
55.14
51.29
42.70
41.50
32.78
29.31
22.14

9b

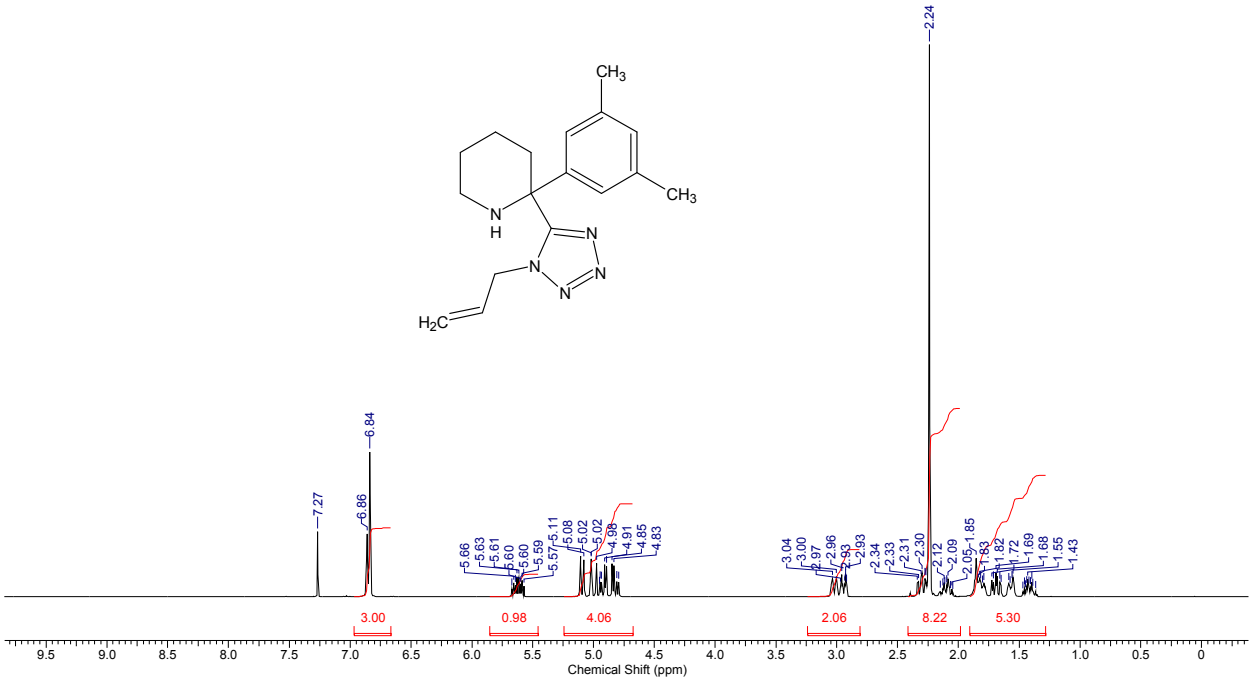

2.21
1.45
CH3
CH3
N
H3C
N
N
N
H3C
CH3
6.59
7.27
6.81
3.05
3.04
3.02
3.02
2.97
2.93
2.93
2.26
2.25
2.19
2.11
2.04
1.59
1.57
1.43
1.42
1.41
1.01
2.00
2.03
9.17
1.14
12.11
Chemical Shift (ppm)

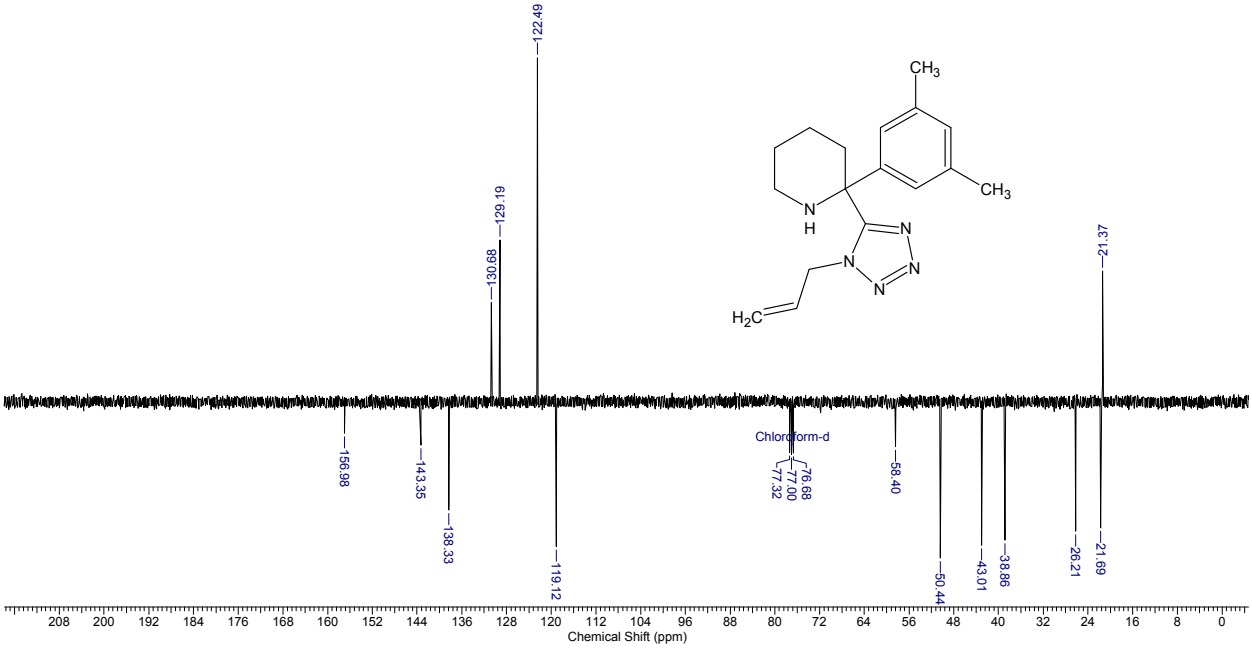

30.09
CH3
CH3
N
H3C
N
N
N
H3C
CH3
128.48
121.95
21.27
Chloroform-d
157.11
146.05
137.91
77.32
77.00
76.68
63.85
60.43
43.41
26.26
22.10
Chemical Shift (ppm)

9c

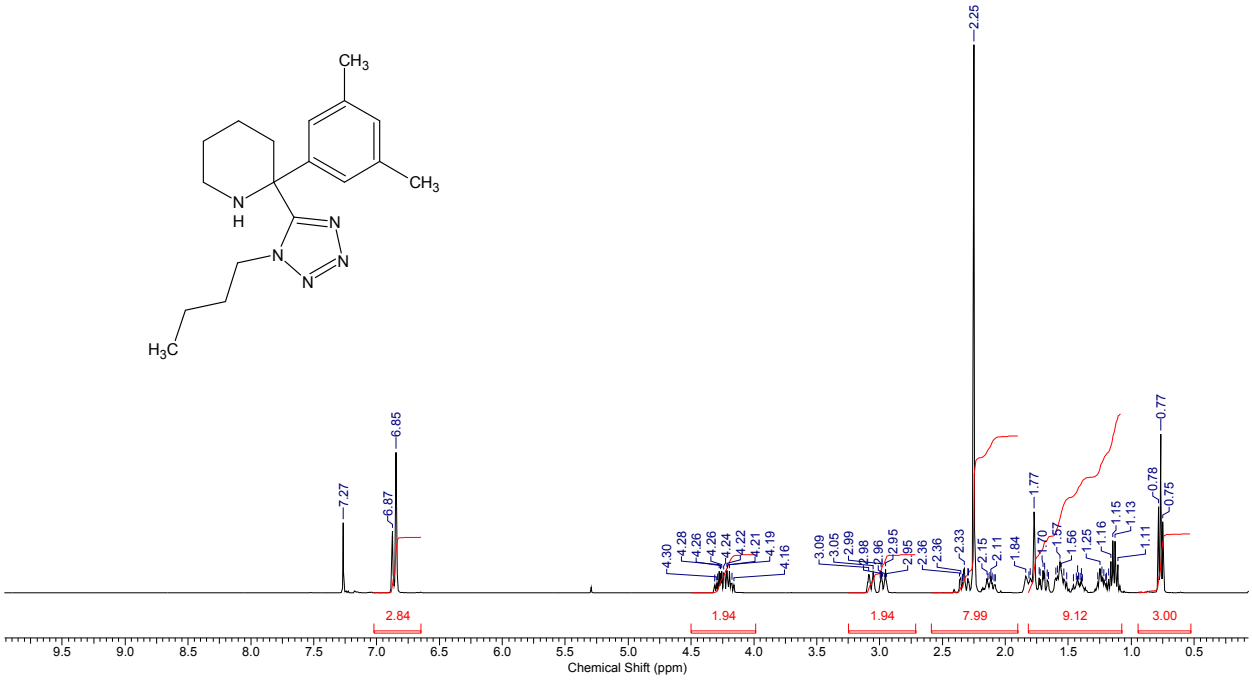

CH3
CH3
N
H
N
N
N
N
H3C
O
O
2.25
1.13
1.14
1.11
6.84
7.27
6.86
5.29
5.25
5.03
4.99
3.03
2.05
1.94
2.03
7.03
1.17
4.23
1.13
2.99
Chemical Shift (ppm)
Chloroform-d
165.67
157.35
142.33
138.41
77.32
77.00
76.68
61.87
58.02
49.04
42.85
38.56
26.36
21.54
129.24
122.36
21.38
13.81
Chemical Shift (ppm)

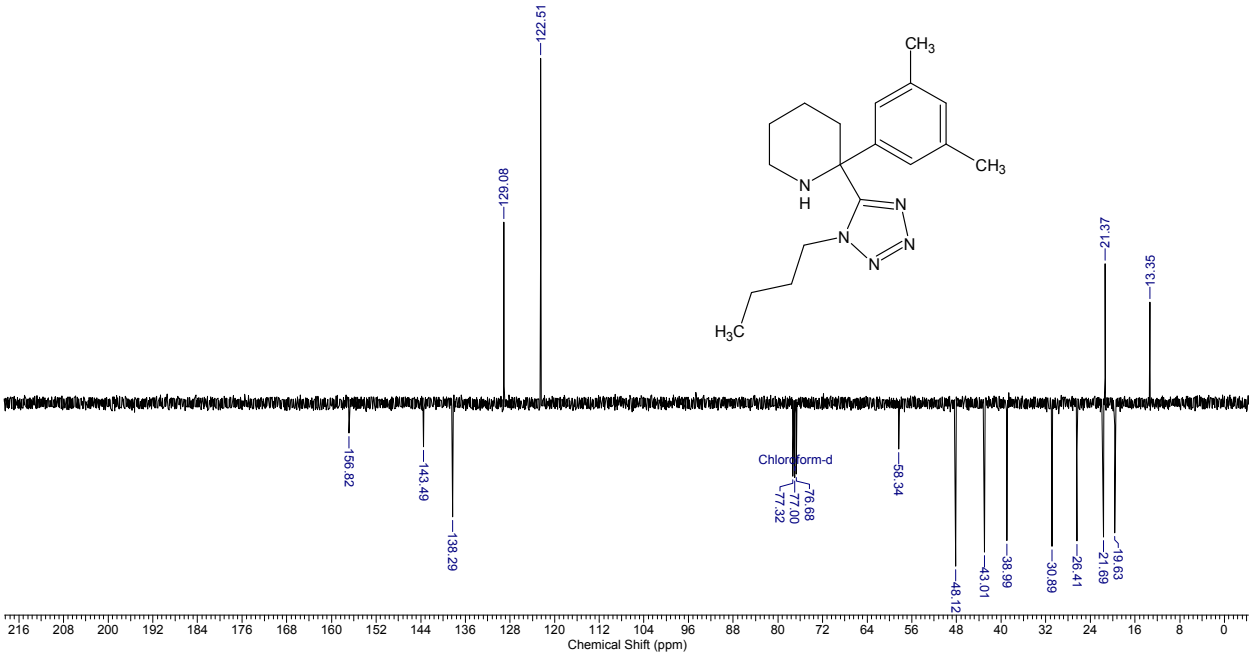
9d


CH3
CH3
N
H
N
N
N
H2C
2.24
6.84
7.27
6.86
5.66
5.63
5.61
5.60
5.59
5.57
5.11
5.08
5.02
5.02
4.98
4.91
4.85
4.83
3.04
3.00
2.97
2.96
2.93
2.34
2.33
2.31
2.30
2.12
2.09
2.05
1.85
1.83
1.82
1.72
1.69
1.68
1.55
1.43
3.00
0.98
4.06
2.06
8.22
5.30
Chemical Shift (ppm)

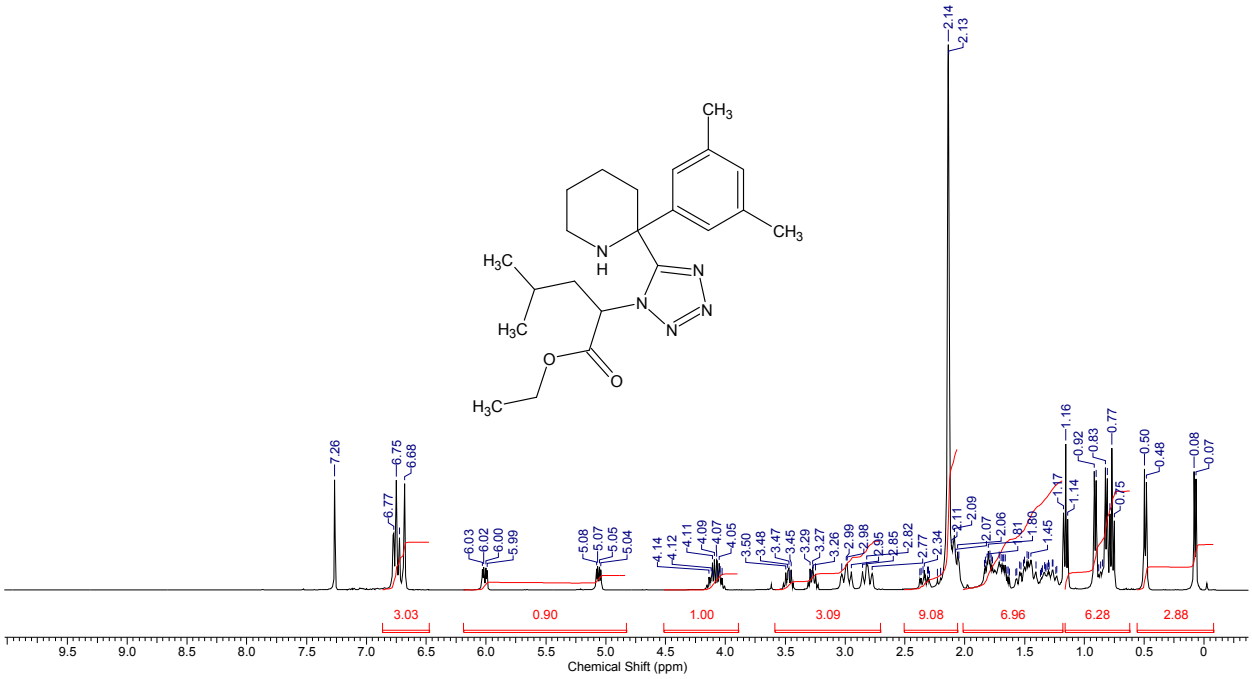

CH3
CH3
N
H
N
N
N
H2C
122.49
129.19
130.68
21.37
156.98
143.35
138.33
119.12
Chloroform-d
77.32
77.00
76.68
58.40
50.44
43.01
38.86
26.21
21.69
Chemical Shift (ppm)

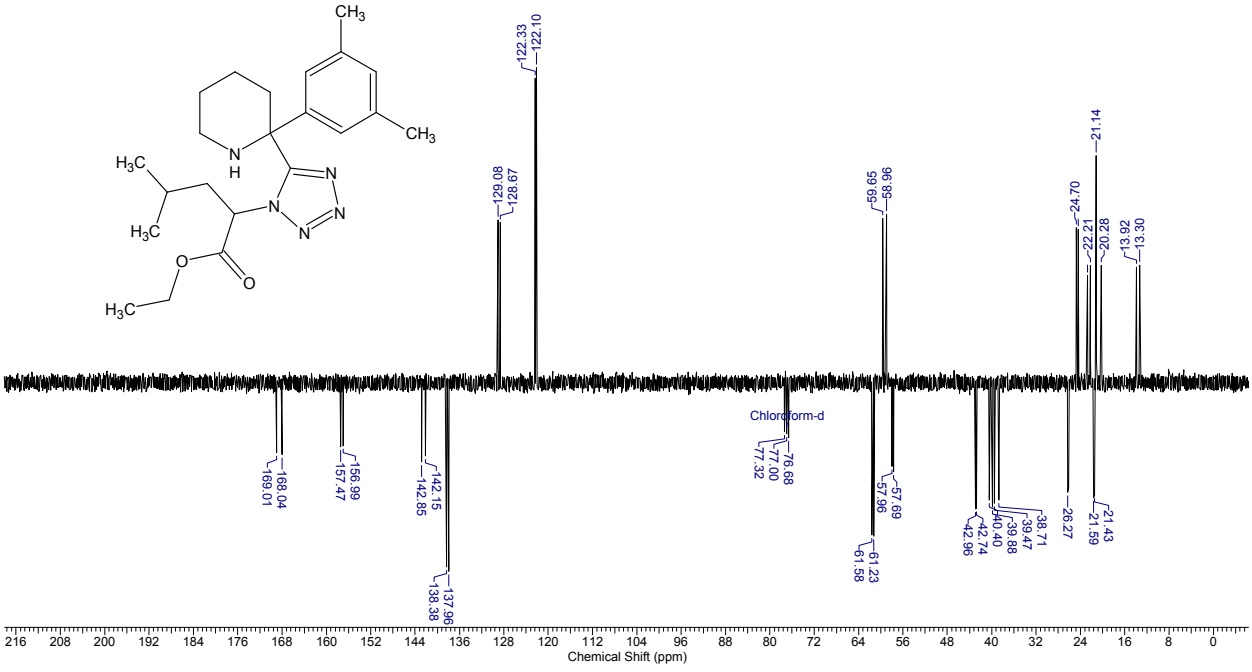
9e


CH3
CH3
N
H
N
N
N
N
H3C
2.25
6.85
7.27
6.87
0.77
0.78
0.75
1.77
4.30
4.28
4.26
4.26
4.24
4.22
4.21
4.19
4.16
3.09
3.05
2.99
2.98
2.96
2.95
2.95
2.36
2.36
2.33
2.15
2.11
1.84
1.70
1.57
1.56
1.25
1.16
1.15
1.13
1.11
2.84
1.94
1.94
7.99
9.12
3.00
Chemical Shift (ppm)

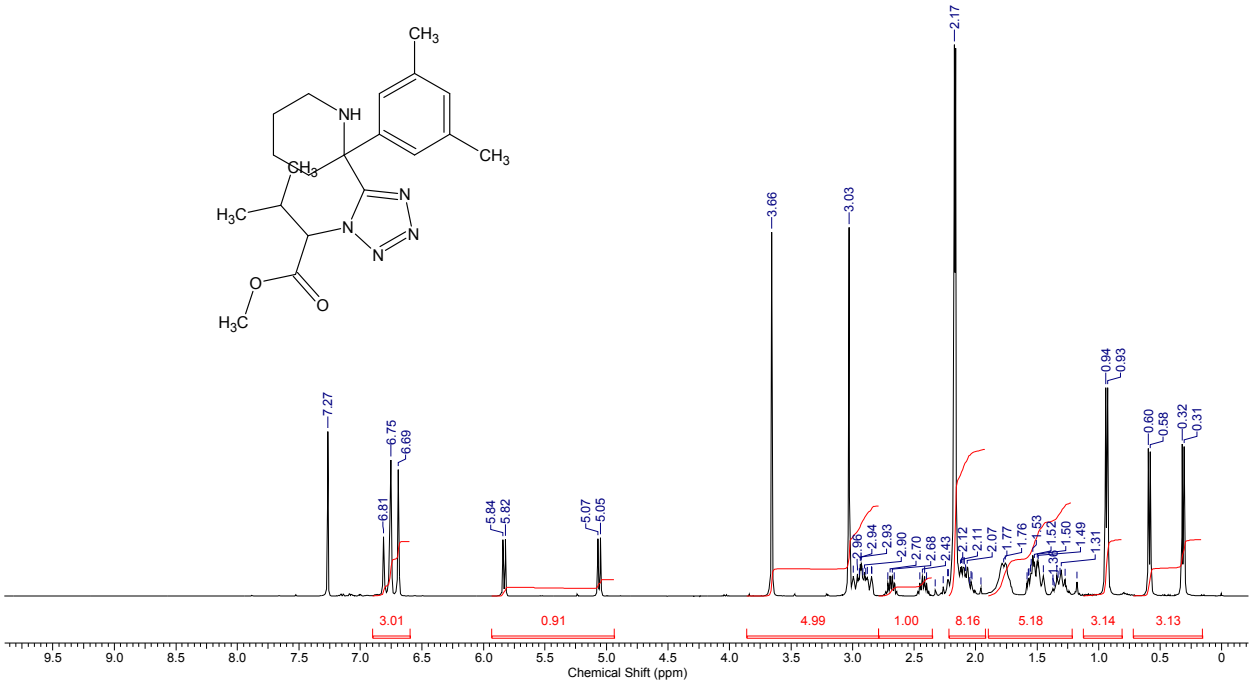

122.51
129.08
CH3
CH3
N
H
N
N
N
N
H3C
21.37
13.35
156.82
143.49
138.29
Chloroform-d
77.32
77.00
76.68
58.34
48.12
43.01
38.99
30.89
26.41
21.69
19.63
Chemical Shift (ppm)

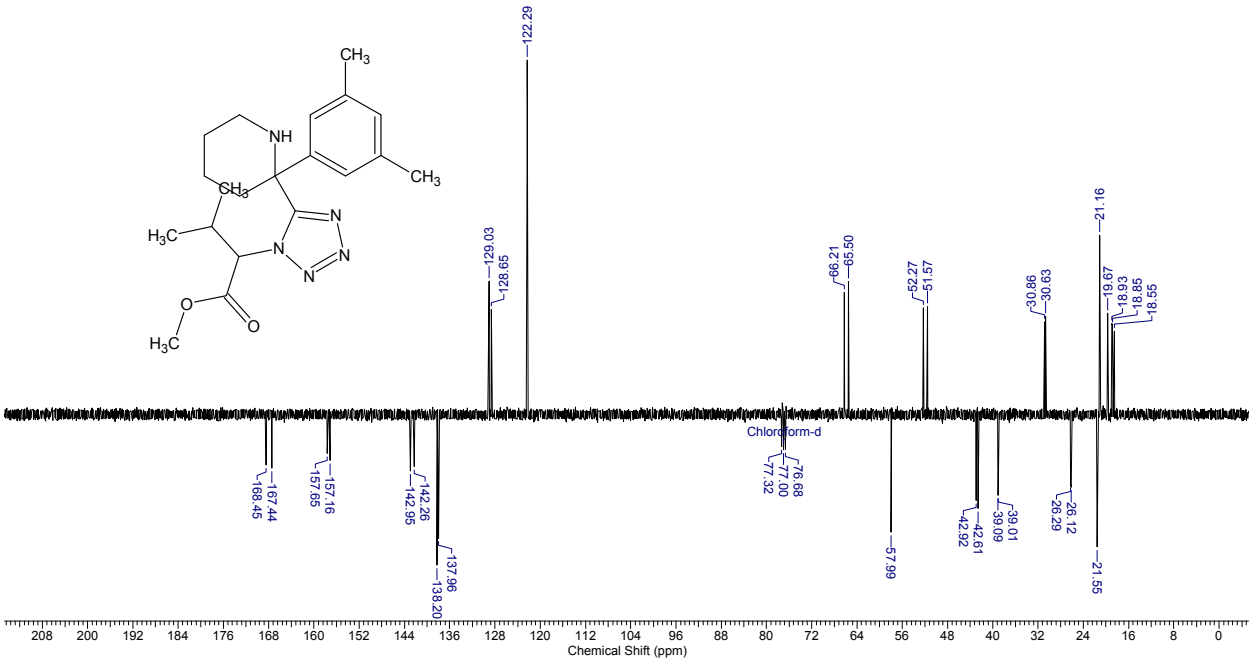
9f


CH3
CH3
N
H
H3C
H3C
N
N
N
O
O
H3C
7.26
6.77
6.75
6.68
6.03
6.02
6.00
5.99
5.08
5.07
5.05
5.04
4.14
4.12
4.11
4.09
4.07
4.05
3.50
3.48
3.47
3.45
3.29
3.27
3.26
2.99
2.98
2.95
2.85
2.82
2.77
2.34
2.14
2.13
2.11
2.09
2.07
2.06
1.81
1.80
1.45
1.17
1.16
1.14
0.92
0.83
0.77
0.76
0.50
0.48
0.08
0.07
3.03
0.90
1.00
3.09
9.08
6.96
6.28
2.88
Chemical Shift (ppm)

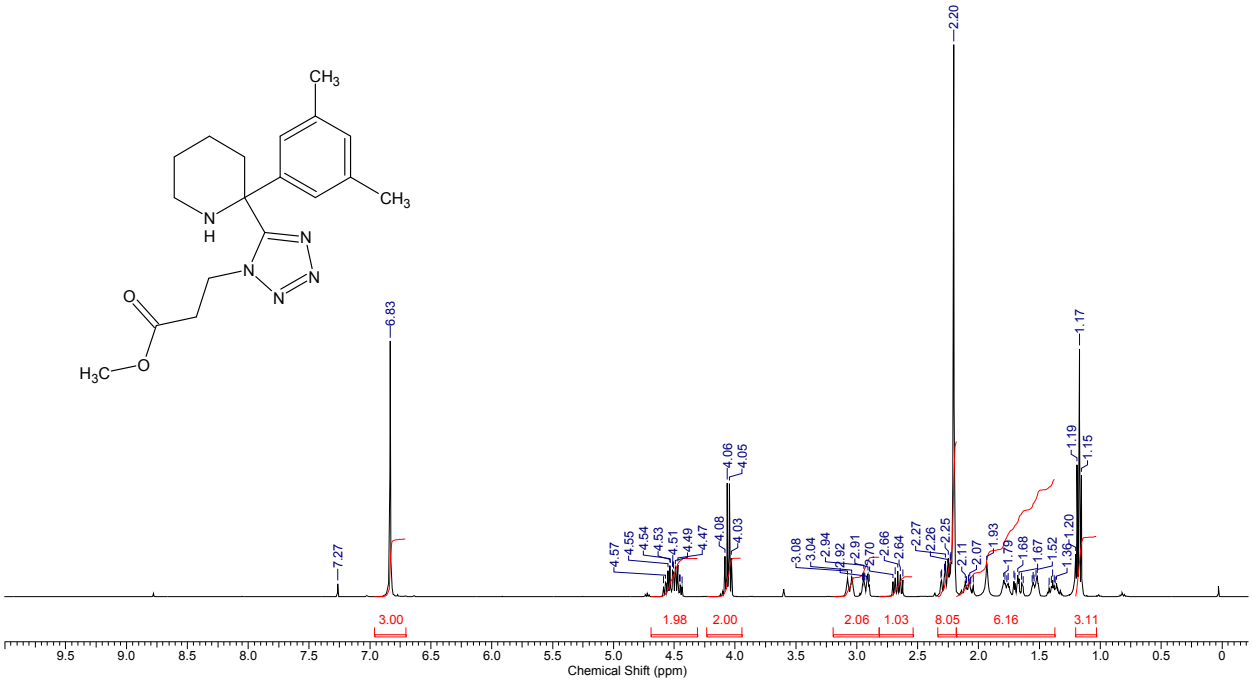

CH3
CH3
N
H
H3C
H3C
N
N
N
O
O
H3C
122.33
122.10
129.08
128.67
59.69
58.96
21.14
24.70
22.21
20.28
13.92
13.30
Chloroform-d
77.32
77.00
76.68
57.96
57.69
61.58
61.23
42.96
42.74
40.40
39.88
39.47
38.71
26.27
21.59
21.43
169.01
168.04
157.47
156.99
142.85
142.15
138.38
137.96
Chemical Shift (ppm)

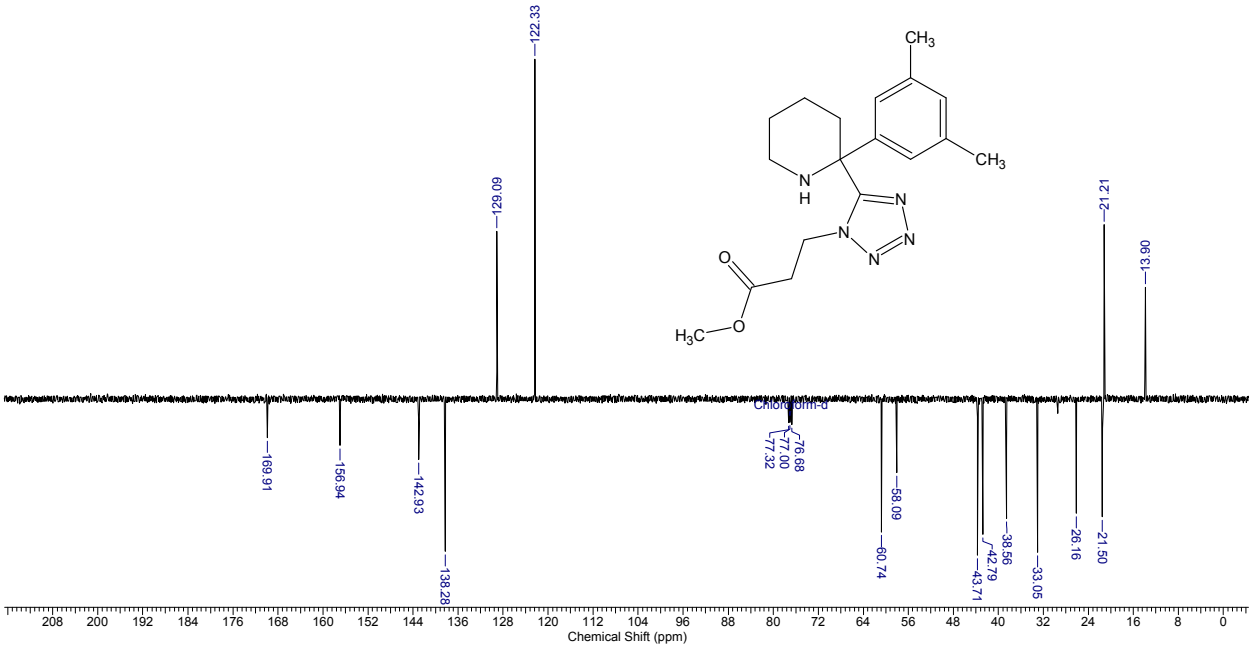
9g


CH3
NH
CH3
CH3
H3C
N
N
N
N
O
O
H3C
2.17
3.66
3.03
7.27
6.75
6.69
6.81
5.84
5.82
5.07
5.05
0.94
0.93
0.60
0.58
0.32
0.31
3.01
0.91
4.99
1.00
8.16
5.18
3.14
3.13
Chemical Shift (ppm)

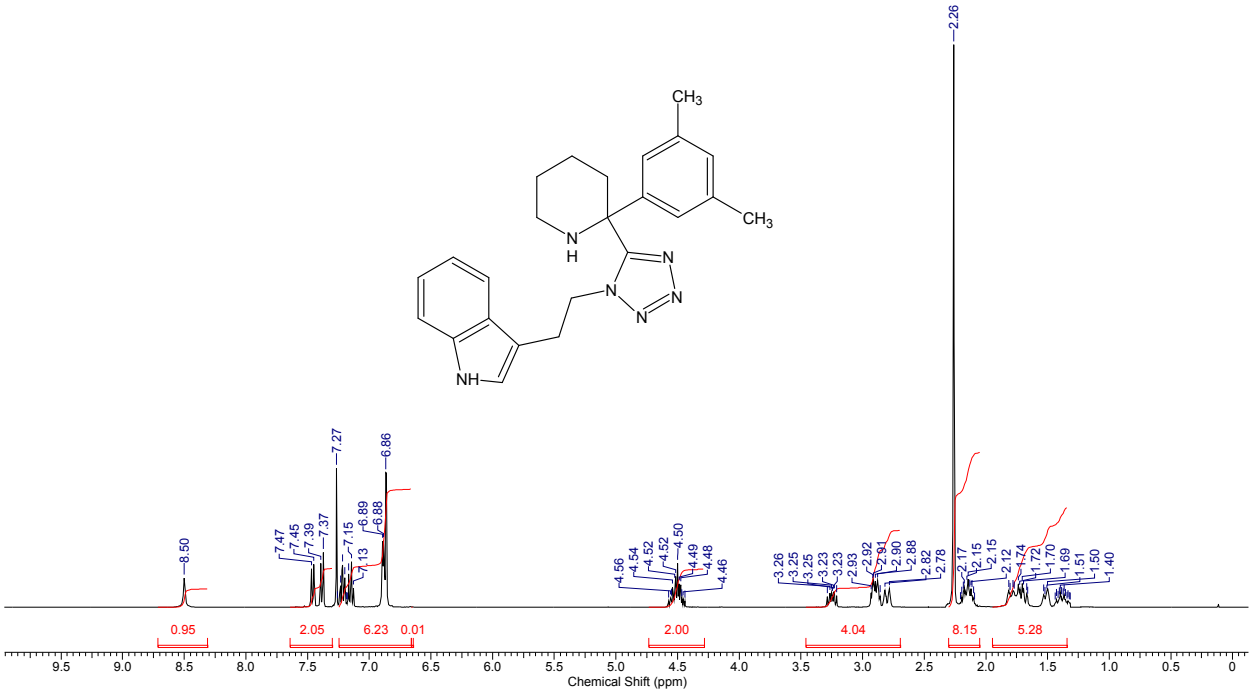

CH3
NH
CH3
CH3
H3C
N
N
N
N
O
O
H3C
122.29
129.03
128.65
66.21
65.50
52.27
51.57
30.86
30.63
21.16
19.67
18.93
18.85
18.55
Chloroform-d
168.45
167.44
157.65
157.16
142.95
142.26
138.20
137.96
77.32
77.00
76.68
57.99
42.92
42.61
39.09
39.01
26.29
26.12
21.55
Chemical Shift (ppm)

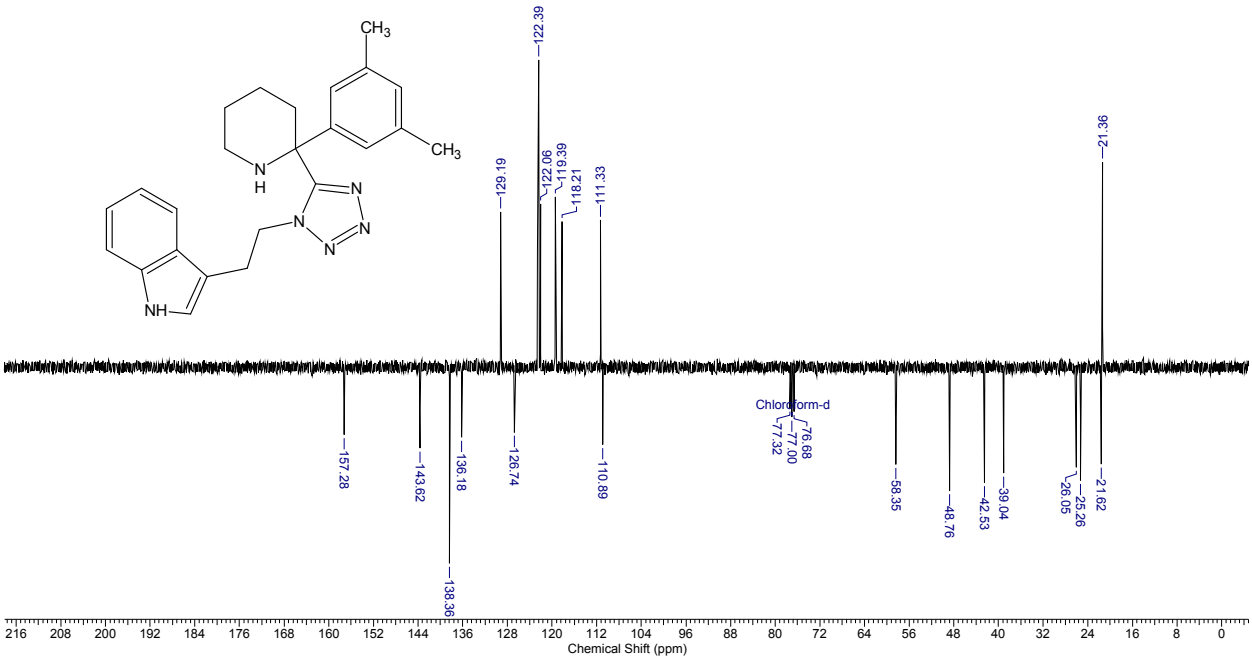
9h


CH3
CH3
N
H
N
N
N
O
O
H3C
7.27
6.83
4.57
4.55
4.54
4.53
4.51
4.49
4.47
4.08
4.06
4.05
4.03
3.08
3.04
2.94
2.92
2.91
2.70
2.66
2.64
2.27
2.26
2.25
2.20
2.11
2.07
1.93
1.79
1.68
1.67
1.52
1.39
1.20
1.19
1.17
1.15
3.00
1.98
2.00
2.06
1.03
8.05
6.16
3.11
Chemical Shift (ppm)

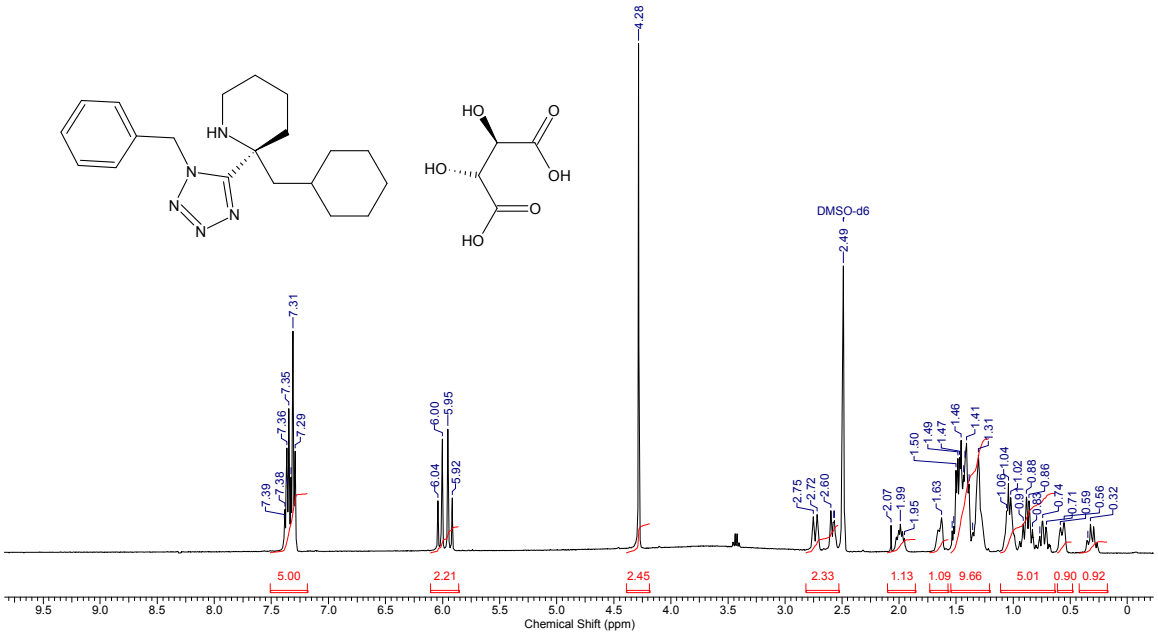

CH3
CH3
N
H
N
N
N
O
O
H3C
129.09
122.33
21.21
13.90
169.91
156.94
142.93
138.28
77.32
77.00
76.68
60.74
58.09
43.71
42.79
38.56
33.05
26.16
21.50
Chloroform-d
Chemical Shift (ppm)

9i

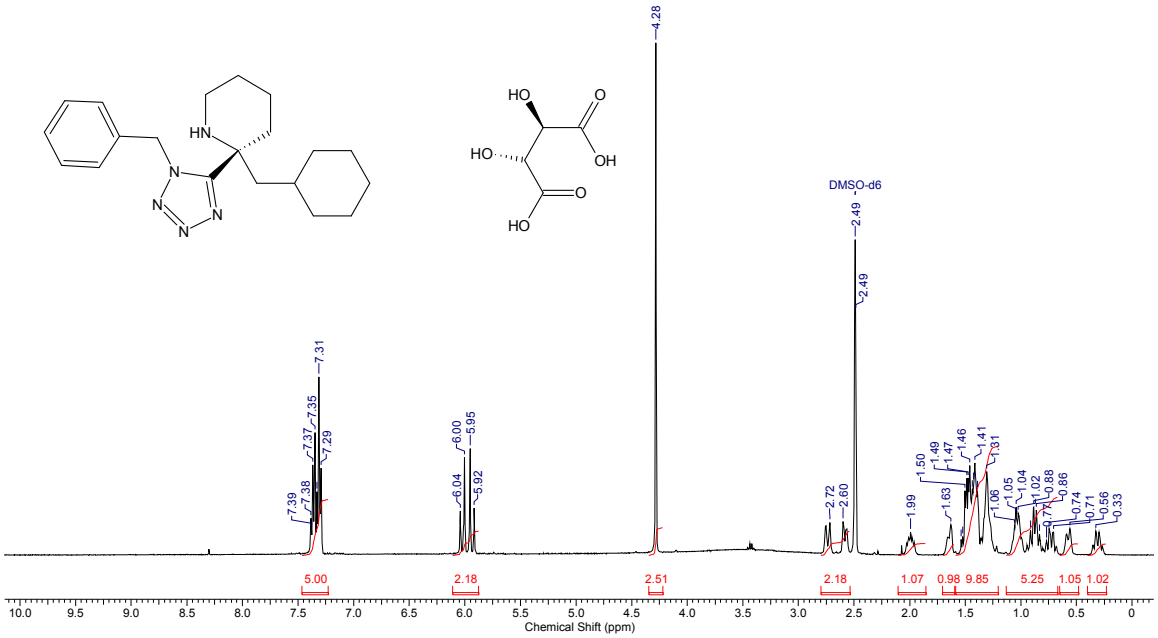

CH3
CH3
N
H
N
N
N
NH
8.50
7.47
7.45
7.39
7.37
7.27
7.15
7.13
6.89
6.88
6.86
4.56
4.54
4.52
4.50
4.49
4.48
4.46
3.26
3.25
3.25
3.23
3.23
2.93
2.92
2.91
2.90
2.88
2.82
2.78
2.26
2.17
2.15
2.15
2.12
1.74
1.72
1.70
1.69
1.51
1.50
1.40
0.95
2.05
6.23
0.01
2.00
4.04
8.15
5.28
Chemical Shift (ppm)



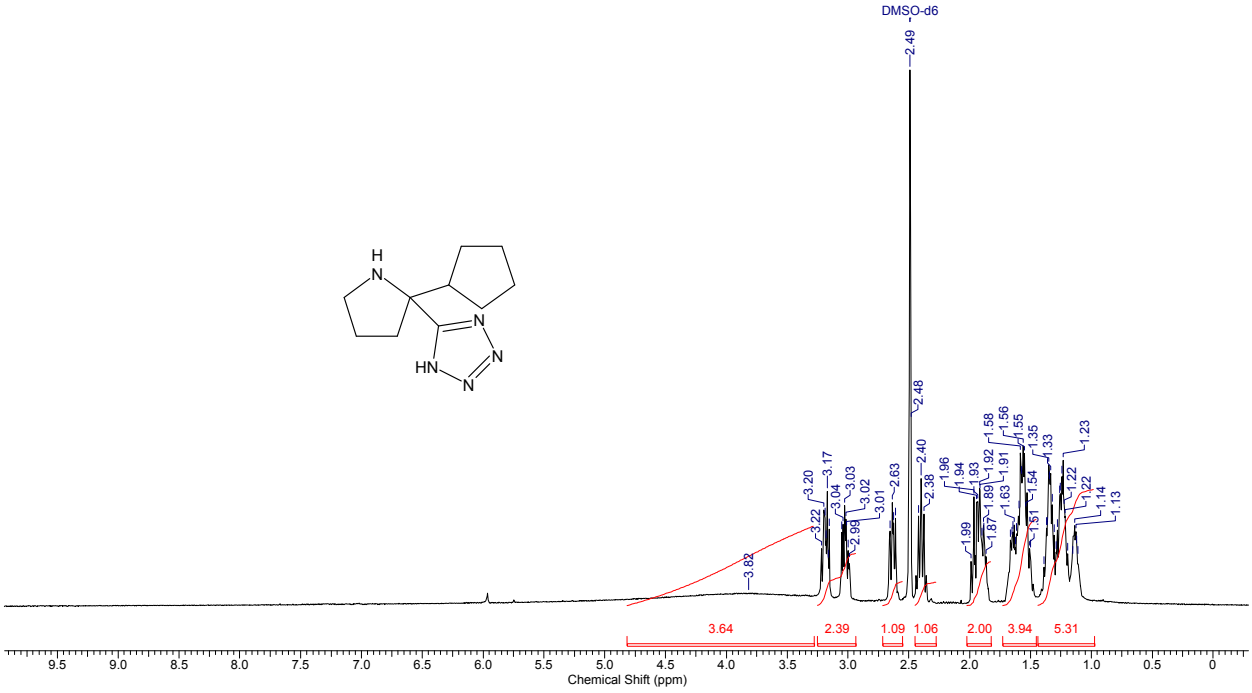
6e R-tartrate

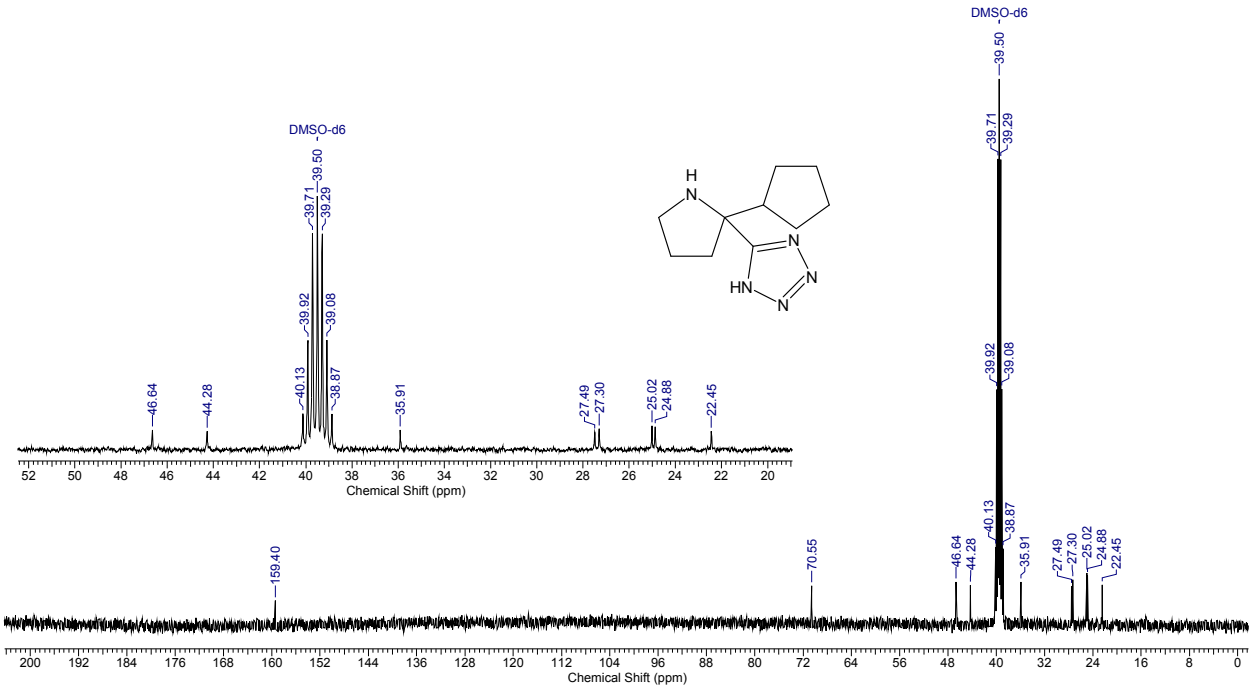


6e S-tartrate

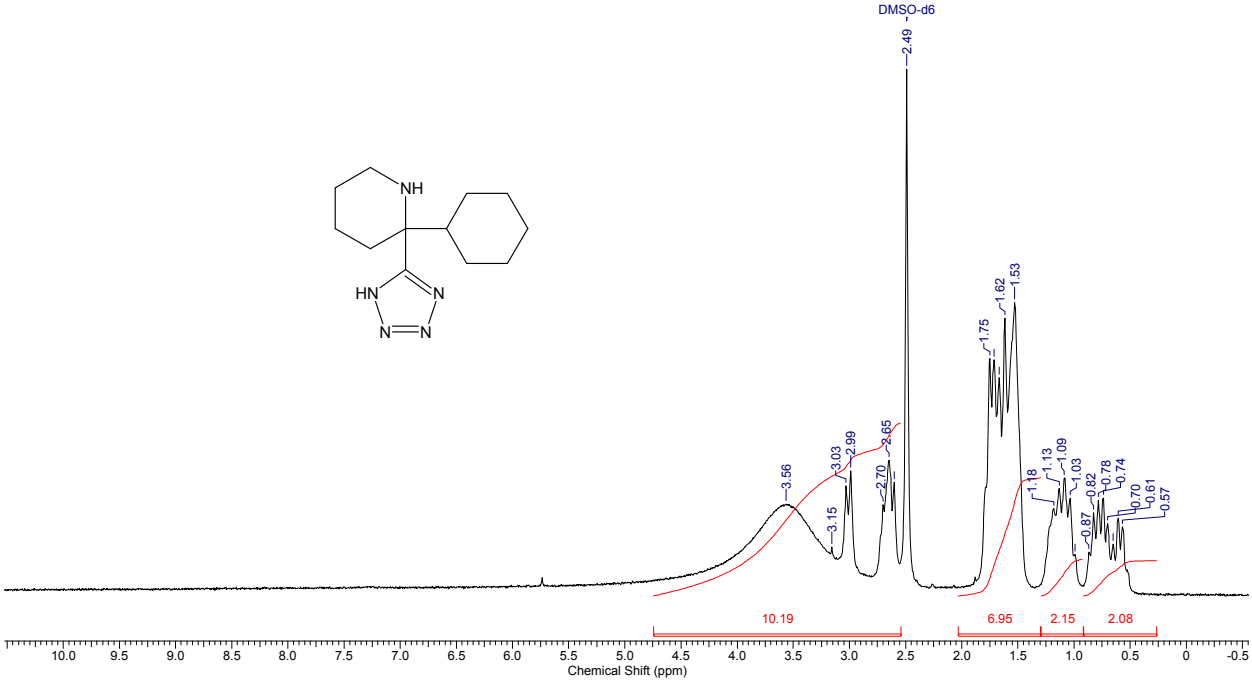


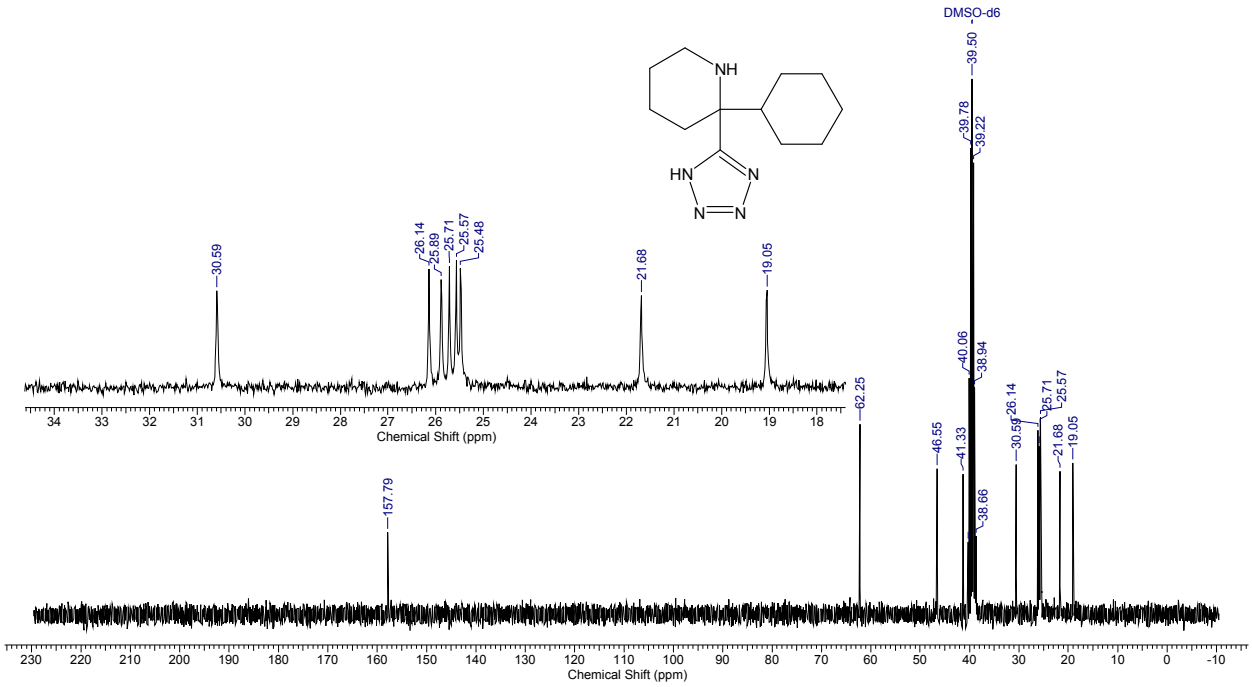
10a



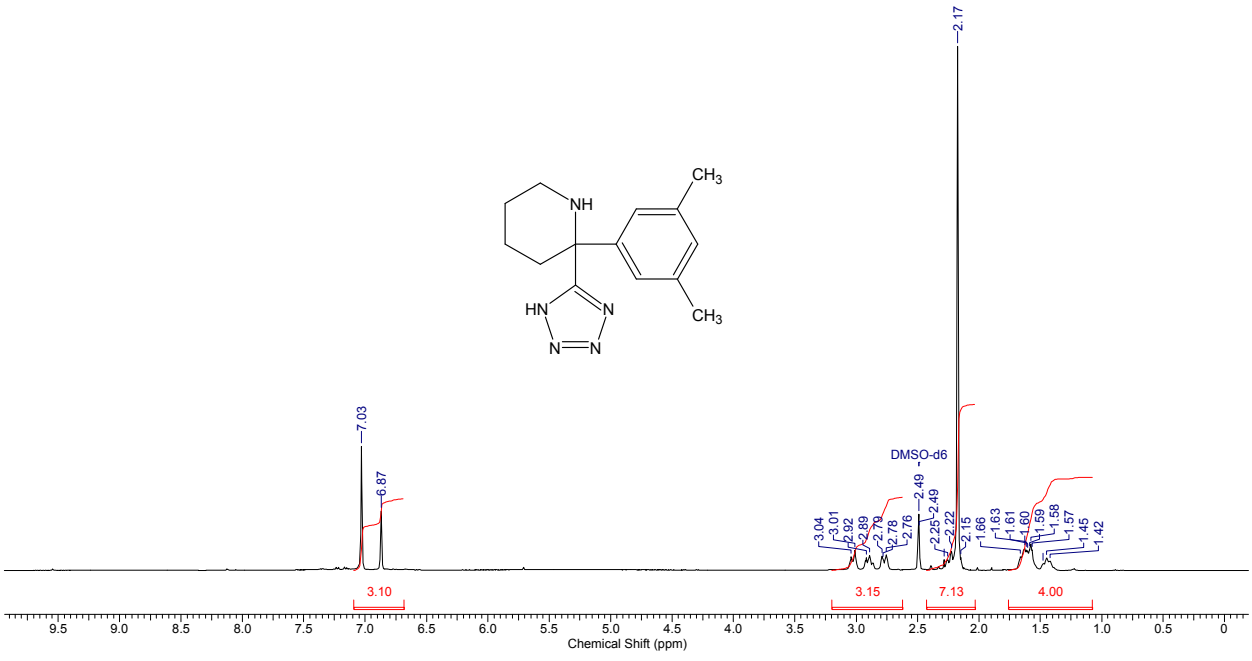

DMSO-d6
39.50
39.71
39.29
39.92
39.08
40.13
38.87
46.64
44.28
35.91
27.49
27.30
25.02
24.88
22.45
159.40
70.55
Chemical Shift (ppm)

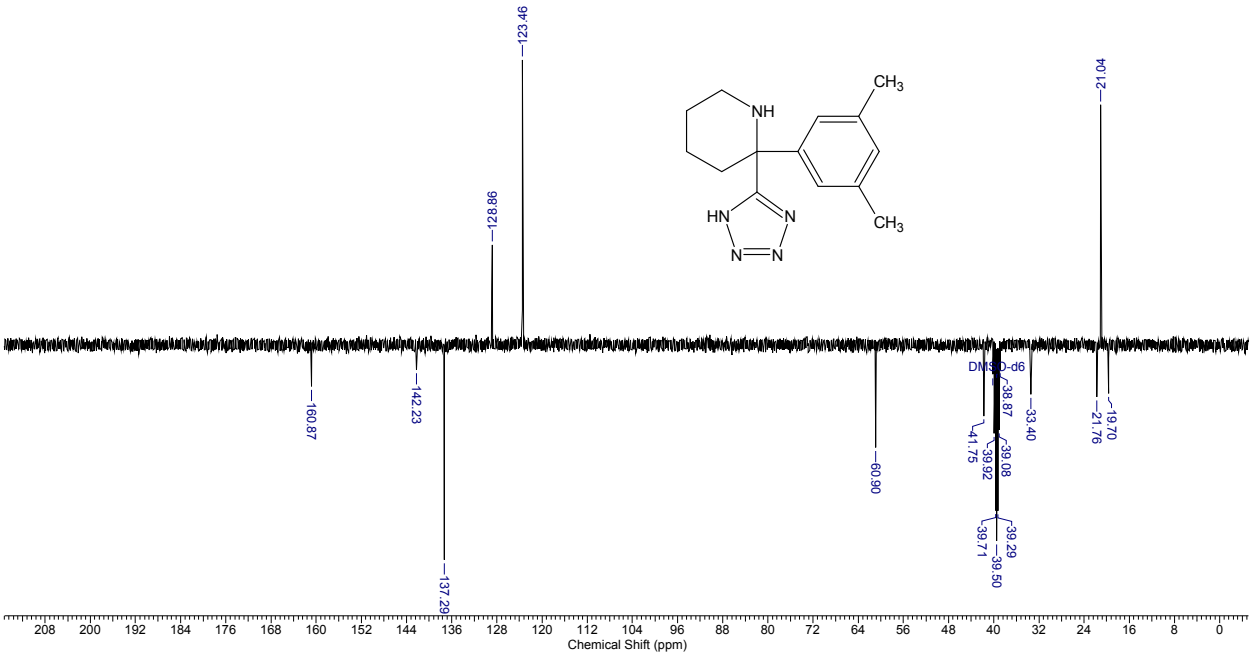
10b


DMSO-d6
2.49
3.56
3.15
3.03
2.99
2.70
2.65
1.75
1.62
1.53
1.18
1.13
1.09
1.03
0.87
0.82
0.78
0.74
0.70
0.61
0.57
10.19
6.95
2.15
2.08
Chemical Shift (ppm)

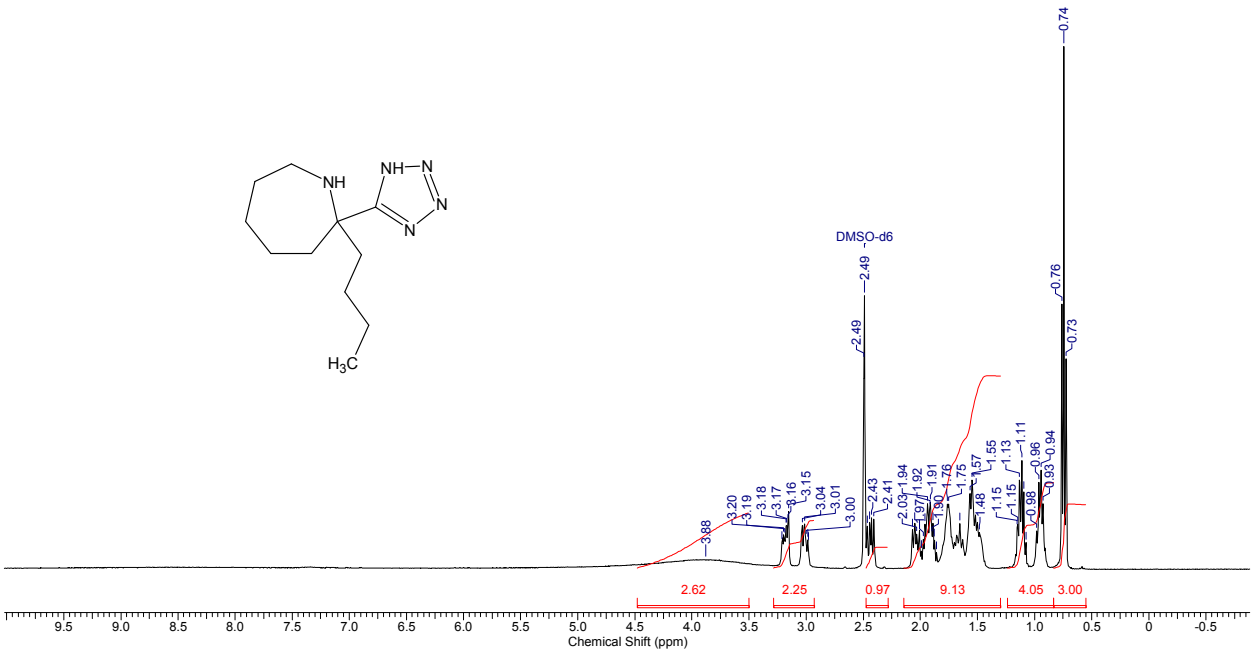

DMSO-d6
NH
HN
N
N
N
39.50
39.78
39.22
30.59
26.14
25.89
25.71
25.57
25.48
21.68
19.05
40.06
38.94
62.25
46.55
41.33
26.14
25.71
25.57
30.59
21.68
19.05
38.66
157.79
Chemical Shift (ppm)
Chemical Shift (ppm)

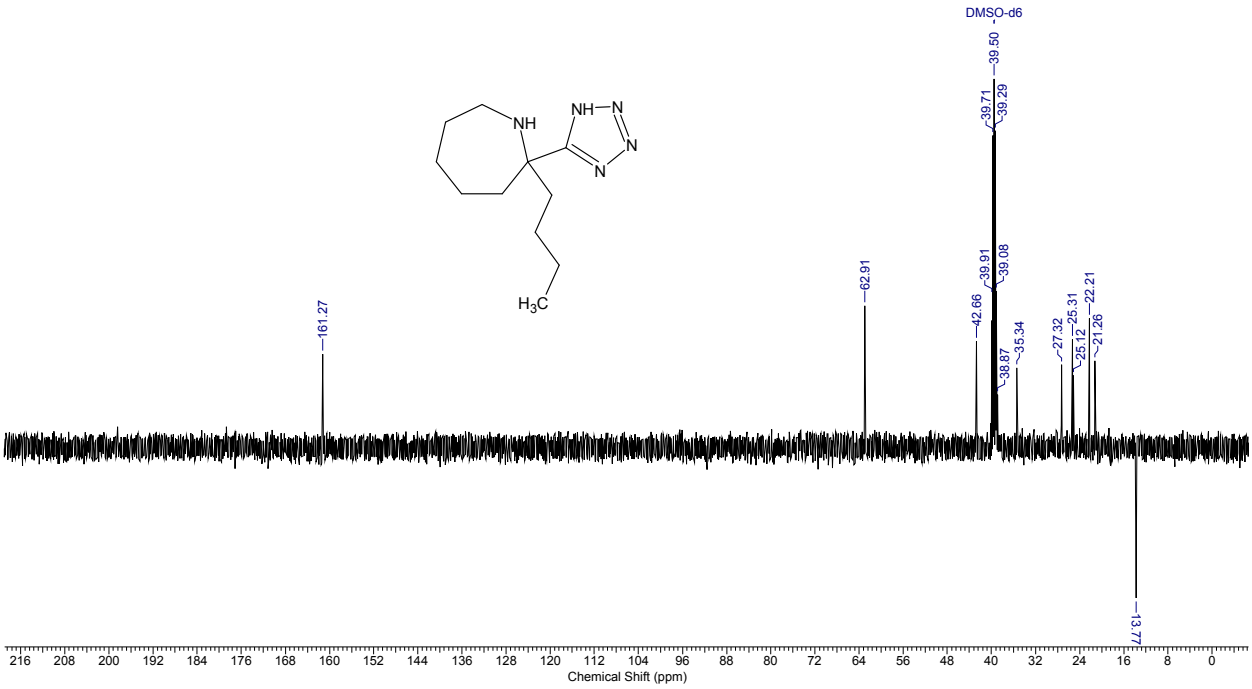
10c


2.17
CH3
NH
HN
N
N
N
CH3
7.03
6.87
DMSO-d6
2.49
3.04
3.01
2.92
2.89
2.79
2.78
2.76
2.25
2.49
2.22
2.15
1.66
1.63
1.61
1.60
1.59
1.58
1.57
1.45
1.42
3.10
3.15
7.13
4.00
Chemical Shift (ppm)

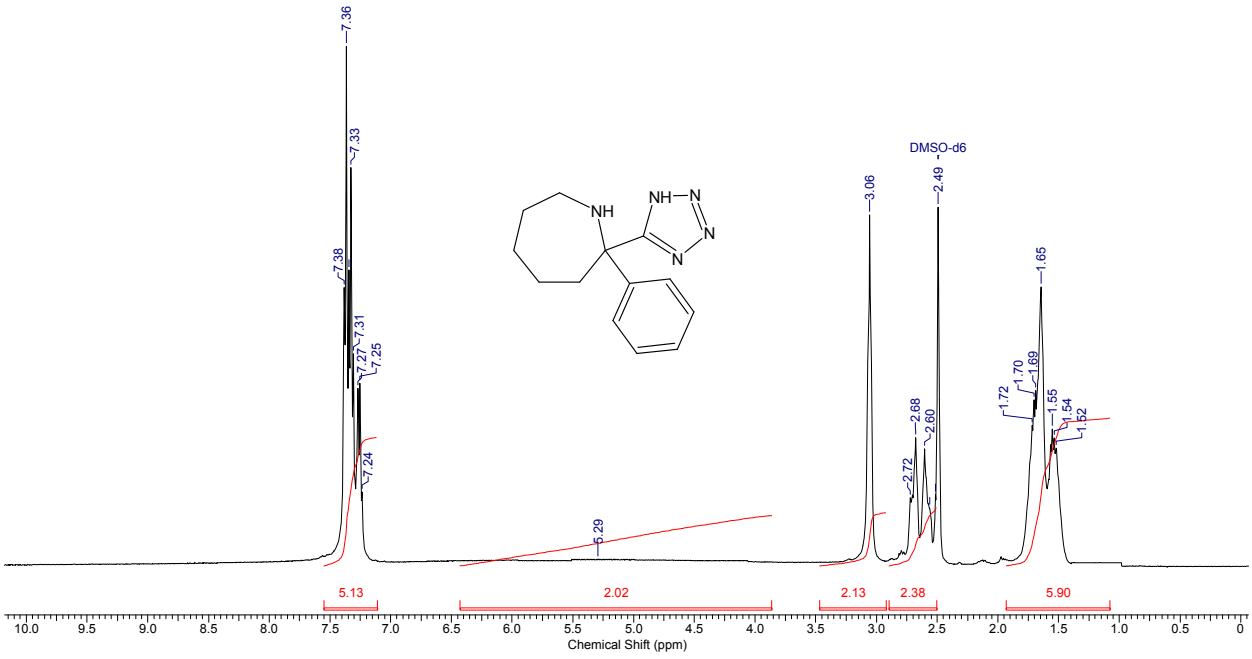

123.46
128.86
21.04
NH
CH3
CH3
HN
N
N
N
160.87
142.23
137.29
60.90
DMSO-d6
41.75
39.92
39.08
38.87
39.71
39.50
39.29
33.40
21.76
19.70
208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)

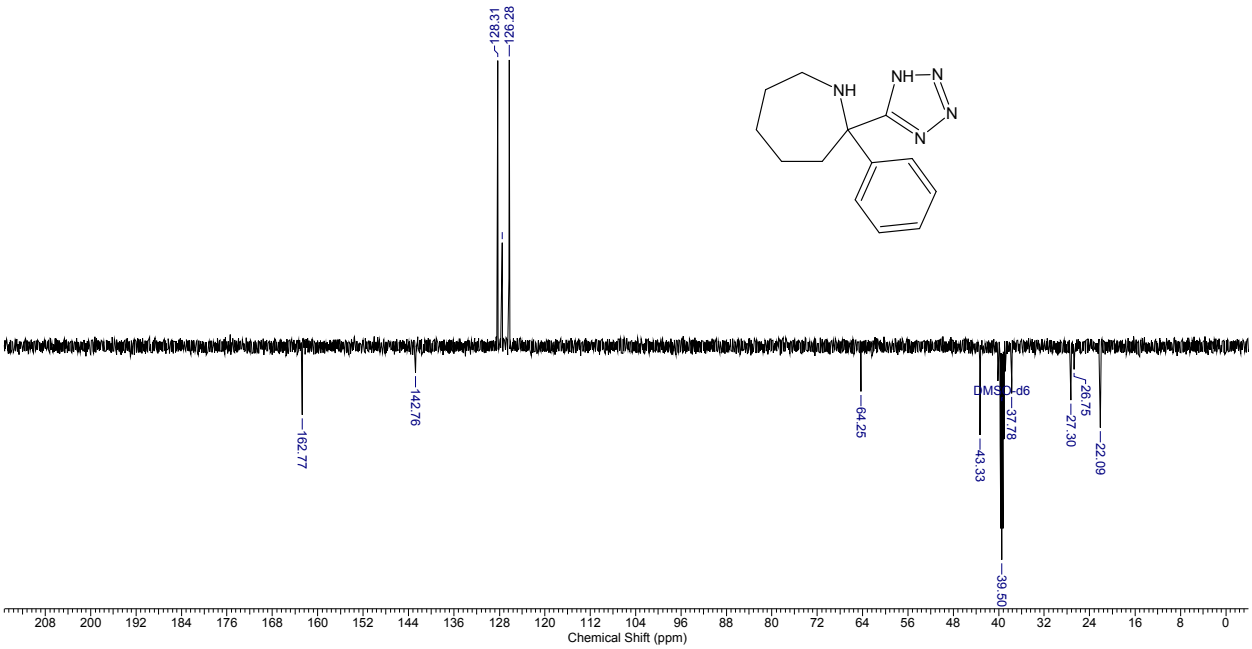
10d


NH
NH
N
N
N
H3C
0.74
DMSO-d6
2.49
2.49
0.76
0.73
3.88
3.20
3.19
3.18
3.17
3.16
3.15
3.04
3.01
3.00
2.43
2.41
2.03
1.94
1.92
1.91
1.76
1.75
1.57
1.55
1.48
1.15
1.13
1.11
0.98
0.96
0.94
0.93
2.62
2.25
0.97
9.13
4.05
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0 -0.5
Chemical Shift (ppm)

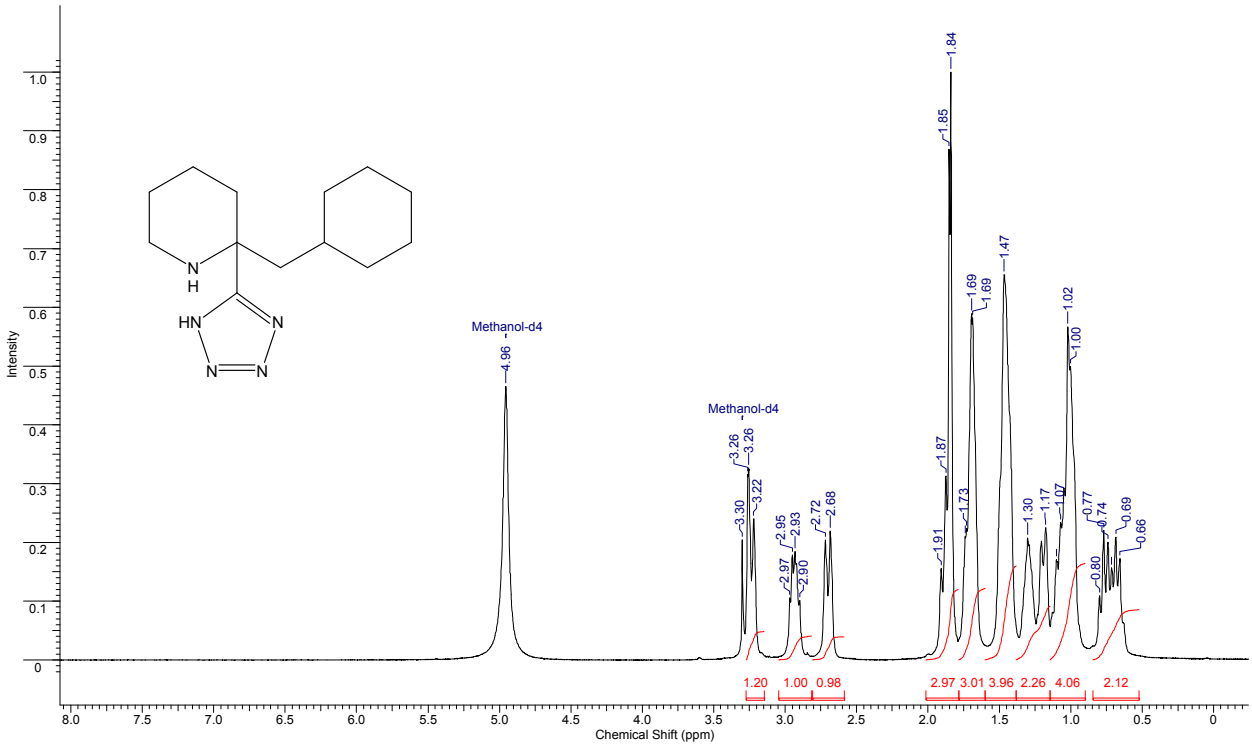

DMSO-d6
39.50
39.71
39.29
39.91
39.08
62.91
42.66
161.27
35.34
38.87
27.32
25.31
25.12
22.21
21.26
13.77
Chemical Shift (ppm)

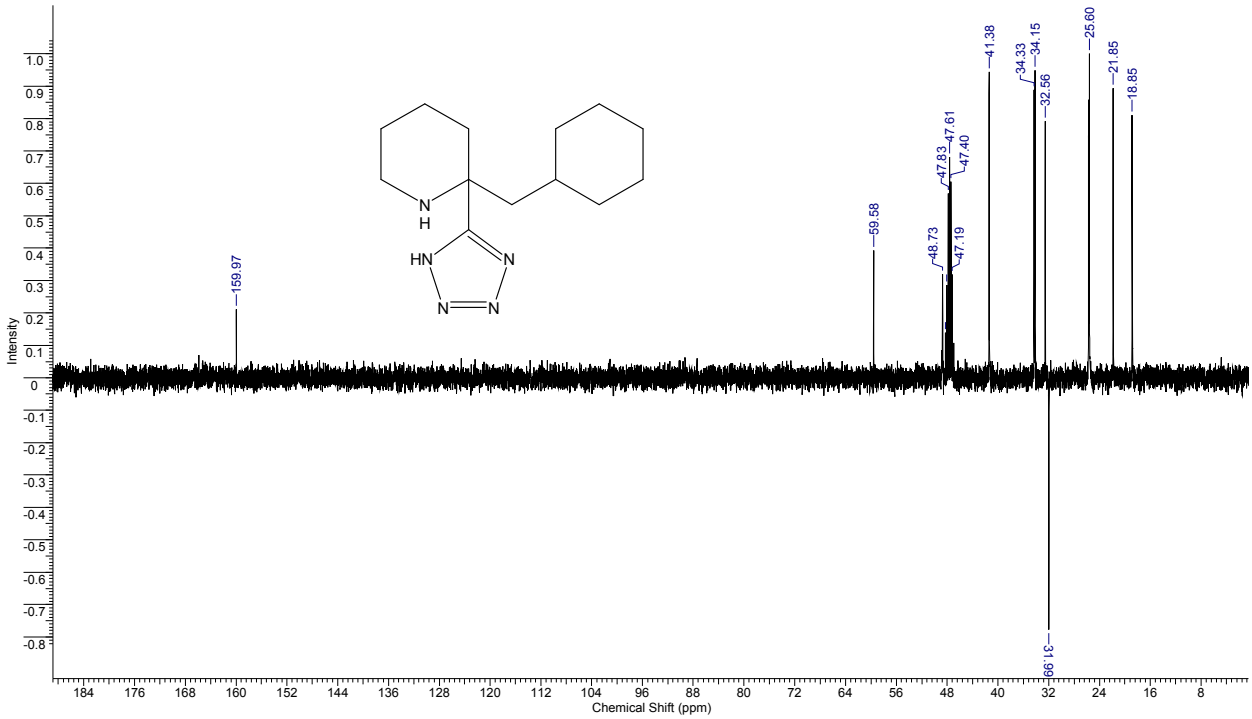
10e


7.36
7.33
7.38
7.31
7.27
7.25
7.24
6.29
3.06
DMSO-d6
2.49
2.72
2.68
2.60
1.65
1.72
1.70
1.69
1.55
1.54
1.52
5.13
2.02
2.13
2.38
5.90
Chemical Shift (ppm)

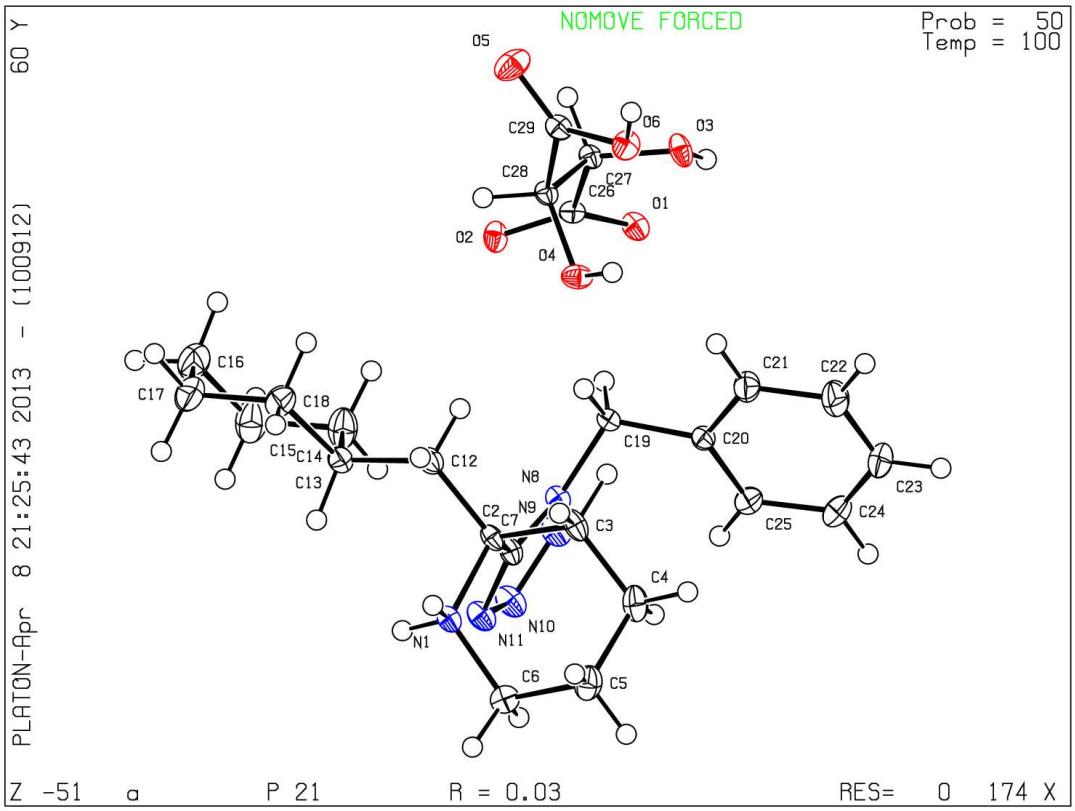

128.31
126.28
NH
NH—N
N
N
162.77
142.76
64.25
DMSO-d6
43.33
39.50
37.78
26.75
27.30
22.09
Chemical Shift (ppm)

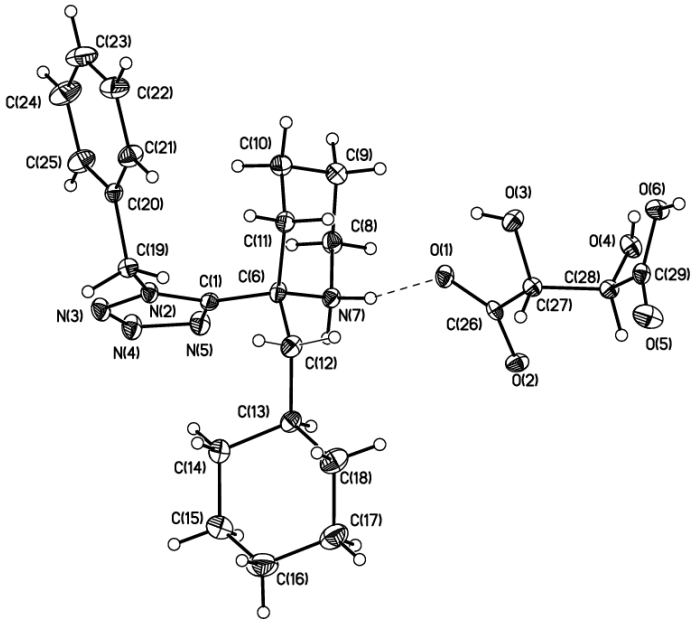
10f


N
H
HN
N
N
N
Intensity
Methanol-d4
4.96
Methanol-d4
3.26
3.26
3.30
3.22
2.95
2.93
2.97
2.90
2.72
2.68
1.84
1.85
1.87
1.91
1.69
1.69
1.73
1.47
1.30
1.17
1.07
1.02
1.00
0.77
0.74
0.80
0.69
0.66
1.20
1.00
0.98
2.97
3.01
3.96
2.26
4.06
2.12
Chemical Shift (ppm)


159.97
59.58
48.73
47.83
47.61
47.40
47.19
41.38
34.33
34.15
32.56
31.99
25.60
21.85
18.85
Intensity
Chemical Shift (ppm)

2. X-Ray structure of tartrate salt of R-**6e**





(ORTEP) The general view of tartrate salt of R-**6e** in representation of atoms by thermal ellipsoids (p=50%).

```
data_a

_audit_creation_method            SHELXL-97
_chemical_name_systematic
;
 ?
;
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety
 'C20 H30 N5, C4 H5 O6'
_chemical_formula_sum
 'C24 H35 N5 O6'
_chemical_formula_weight          489.57

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0033   0.0016
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0106   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            Monoclinic
_symmetry_space_group_name_H-M    'P 21'
_symmetry_space_group_name_Hall   'P 2yb'
_chemical_absolute_configuration   ' rm'

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, y+1/2, -z'

_cell_length_a                    7.5027(2)
_cell_length_b                    20.0377(6)
_cell_length_c                    8.6994(3)
_cell_angle_alpha                 90.00
_cell_angle_beta                  104.1532(5)
_cell_angle_gamma                 90.00
_cell_volume                      1268.14(7)
_cell_formula_units_Z             2
_cell_measurement_temperature     100(2)
_cell_measurement_reflns_used     909
_cell_measurement_theta_min       3
_cell_measurement_theta_max       30
```

```
_exptl_crystal_description        prism
_exptl_crystal_colour             colorless
_exptl_crystal_size_max           0.23
_exptl_crystal_size_mid           0.21
_exptl_crystal_size_min           0.18
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.282
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              524
_exptl_absorpt_coefficient_mu     0.093
_exptl_absorpt_correction_type    multi-scan
_exptl_absorpt_correction_T_min   0.979
_exptl_absorpt_correction_T_max   0.983
_exptl_absorpt_process_details    'SADABS'

_exptl_special_details
;
 ?
;

_diffrn_ambient_temperature       100(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_radiation_monochromator   graphite
_diffrn_measurement_device_type   'Bruker APEX  DUO area detector'
_diffrn_measurement_method        'omega scans'
_diffrn_detector_area_resol_mean  ?
_diffrn_reflns_number             16782
_diffrn_reflns_av_R_equivalents   0.0247
_diffrn_reflns_av_sigmaI/netI     0.0197
_diffrn_reflns_limit_h_min        -10
_diffrn_reflns_limit_h_max        10
_diffrn_reflns_limit_k_min        -28
_diffrn_reflns_limit_k_max        28
_diffrn_reflns_limit_l_min        -12
_diffrn_reflns_limit_l_max        12
_diffrn_reflns_theta_min          2.03
_diffrn_reflns_theta_max          30.00
_reflns_number_total              3806
_reflns_number_gt                 3635
_reflns_threshold_expression      'I>2\s(I)'

_computing_data_collection        'Bruker APEX II'
_computing_cell_refinement        'Bruker SAINT'
_computing_data_reduction         'Bruker SAINT'
_computing_structure_solution     'SHELXS-97 (Sheldrick, 1997)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics     'Bruker SHELXTL'
_computing_publication_material   'Bruker SHELXTL'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR
and
 goodness of fit S are based on F^2^, conventional R-factors R are
based
```

```
 on F, with F set to zero for negative F^2^. The threshold expression
of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc.
and is
 not relevant to the choice of reflections for refinement.  R-factors
based
 on F^2^ are statistically about twice as large as those based on F,
and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0480P)^2^+0.1193P] where
P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_abs_structure_details
 'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack    0.3(6)
_refine_ls_number_reflns          3806
_refine_ls_number_parameters      323
_refine_ls_number_restraints      1
_refine_ls_R_factor_all           0.0311
_refine_ls_R_factor_gt            0.0289
_refine_ls_wR_factor_ref          0.0755
_refine_ls_wR_factor_gt           0.0738
_refine_ls_goodness_of_fit_ref    1.035
_refine_ls_restrained_S_all       1.035
_refine_ls_shift/su_max           0.000
_refine_ls_shift/su_mean          0.000

loop_
 _atom_site_label
 _atom_site_type_symbol
 _atom_site_fract_x
 _atom_site_fract_y
 _atom_site_fract_z
 _atom_site_U_iso_or_equiv
 _atom_site_adp_type
 _atom_site_occupancy
 _atom_site_symmetry_multiplicity
 _atom_site_calc_flag
 _atom_site_refinement_flags
 _atom_site_disorder_assembly
 _atom_site_disorder_group
O1 O 0.48586(13) 0.18373(5) 1.14882(12) 0.01707(19) Uani 1 1 d . . .
N1 N 0.19070(15) 0.22442(6) 0.26083(13) 0.0143(2) Uani 1 1 d . . .
H1NB H 0.2728 0.2060 0.2168 0.030(6) Uiso 1 1 d R . .
H1NA H 0.0871 0.2008 0.2349 0.012(4) Uiso 1 1 d R . .
O2 O 0.40667(13) 0.09729(5) 0.98126(13) 0.0180(2) Uani 1 1 d . . .
```

```
C2 C 0.27436(17) 0.21855(7) 0.43773(15) 0.0140(2) Uani 1 1 d . . .
O3 O 0.83532(13) 0.17417(5) 1.15652(12) 0.0186(2) Uani 1 1 d . . .
H3O H 0.7719 0.1963 1.2114 0.039(6) Uiso 1 1 d R . .
C3 C 0.44415(18) 0.26402(8) 0.47572(16) 0.0177(3) Uani 1 1 d . . .
H3A H 0.4989 0.2631 0.5914 0.021 Uiso 1 1 calc R . .
H3B H 0.5366 0.2462 0.4225 0.021 Uiso 1 1 calc R . .
O4 O 0.72387(14) 0.15872(5) 0.81967(12) 0.0178(2) Uani 1 1 d . . .
H4O H 0.8066 0.1781 0.8276 0.038(7) Uiso 1 1 d R . .
C4 C 0.4013(2) 0.33609(8) 0.42365(18) 0.0226(3) Uani 1 1 d . . .
H4A H 0.5154 0.3630 0.4509 0.027 Uiso 1 1 calc R . .
H4B H 0.3134 0.3552 0.4800 0.027 Uiso 1 1 calc R . .
O5 O 1.00821(16) 0.02379(6) 0.99398(16) 0.0263(2) Uani 1 1 d . . .
C5 C 0.3185(2) 0.33864(8) 0.24481(19) 0.0234(3) Uani 1 1 d . . .
H5A H 0.2846 0.3852 0.2125 0.028 Uiso 1 1 calc R . .
H5B H 0.4113 0.3236 0.1886 0.028 Uiso 1 1 calc R . .
O6 O 1.07785(13) 0.12945(5) 0.94406(12) 0.0182(2) Uani 1 1 d . . .
H6O H 1.1834 0.1168 0.9553 0.041(7) Uiso 1 1 d R . .
C6 C 0.1490(2) 0.29444(7) 0.19795(17) 0.0186(3) Uani 1 1 d . . .
H6A H 0.0493 0.3136 0.2406 0.022 Uiso 1 1 calc R . .
H6B H 0.1060 0.2931 0.0810 0.022 Uiso 1 1 calc R . .
C7 C 0.13036(17) 0.24034(7) 0.52213(15) 0.0142(2) Uani 1 1 d . . .
N8 N 0.15763(15) 0.24955(6) 0.67940(13) 0.0140(2) Uani 1 1 d . . .
N9 N -0.00651(16) 0.26568(7) 0.70954(15) 0.0182(2) Uani 1 1 d . . .
N10 N -0.12607(16) 0.26675(7) 0.57411(15) 0.0205(2) Uani 1 1 d . . .
N11 N -0.04546(16) 0.25090(7) 0.45471(14) 0.0182(2) Uani 1 1 d . . .
C12 C 0.33439(18) 0.14515(7) 0.47372(16) 0.0169(3) Uani 1 1 d . . .
H12B H 0.4267 0.1347 0.4105 0.026(5) Uiso 1 1 d R . .
H12A H 0.3993 0.1423 0.5827 0.021(5) Uiso 1 1 d R . .
C13 C 0.1899(2) 0.08897(7) 0.43845(16) 0.0181(3) Uani 1 1 d . . .
H13A H 0.0986 0.0996 0.3368 0.022 Uiso 1 1 calc R . .
C14 C 0.0865(3) 0.08069(9) 0.5691(2) 0.0289(3) Uani 1 1 d . . .
H14A H 0.0161 0.1219 0.5760 0.035 Uiso 1 1 calc R . .
H14B H 0.1765 0.0744 0.6722 0.035 Uiso 1 1 calc R . .
C15 C -0.0462(3) 0.02087(10) 0.5380(2) 0.0347(4) Uani 1 1 d . . .
H15A H -0.1051 0.0160 0.6276 0.042 Uiso 1 1 calc R . .
H15B H -0.1440 0.0291 0.4407 0.042 Uiso 1 1 calc R . .
C16 C 0.0550(3) -0.04360(10) 0.5188(2) 0.0362(4) Uani 1 1 d . . .
H16A H 0.1451 -0.0542 0.6195 0.043 Uiso 1 1 calc R . .
H16B H -0.0341 -0.0809 0.4937 0.043 Uiso 1 1 calc R . .
C17 C 0.1544(3) -0.03617(9) 0.3863(2) 0.0311(4) Uani 1 1 d . . .
H17A H 0.0630 -0.0297 0.2841 0.037 Uiso 1 1 calc R . .
H17B H 0.2238 -0.0775 0.3786 0.037 Uiso 1 1 calc R . .
C18 C 0.2870(2) 0.02326(8) 0.4170(2) 0.0266(3) Uani 1 1 d . . .
H18A H 0.3450 0.0279 0.3268 0.032 Uiso 1 1 calc R . .
H18B H 0.3857 0.0144 0.5135 0.032 Uiso 1 1 calc R . .
C19 C 0.32118(18) 0.24742(7) 0.81326(15) 0.0143(2) Uani 1 1 d . . .
H19A H 0.4118 0.2161 0.7875 0.017 Uiso 1 1 calc R . .
H19B H 0.2854 0.2299 0.9079 0.017 Uiso 1 1 calc R . .
C20 C 0.41090(19) 0.31498(7) 0.85244(15) 0.0150(2) Uani 1 1 d . . .
C21 C 0.6020(2) 0.31835(8) 0.90286(19) 0.0217(3) Uani 1 1 d . . .
H21A H 0.6737 0.2791 0.9055 0.026 Uiso 1 1 calc R . .
C22 C 0.6877(2) 0.37937(9) 0.9494(2) 0.0289(3) Uani 1 1 d . . .
H22A H 0.8179 0.3815 0.9832 0.035 Uiso 1 1 calc R . .
C23 C 0.5847(3) 0.43672(8) 0.9468(2) 0.0305(4) Uani 1 1 d . . .
H23A H 0.6435 0.4782 0.9790 0.037 Uiso 1 1 calc R . .
C24 C 0.3948(3) 0.43322(8) 0.8969(2) 0.0308(4) Uani 1 1 d . . .
H24A H 0.3236 0.4726 0.8950 0.037 Uiso 1 1 calc R . .
```

```
C25 C 0.3070(2) 0.37275(8) 0.84935(19) 0.0229(3) Uani 1 1 d . . .
H25A H 0.1768 0.3709 0.8150 0.027 Uiso 1 1 calc R . .
C26 C 0.52063(17) 0.13529(7) 1.06981(15) 0.0129(2) Uani 1 1 d . . .
C27 C 0.72443(16) 0.11984(7) 1.08525(15) 0.0127(2) Uani 1 1 d . . .
H27A H 0.7582 0.0799 1.1554 0.015 Uiso 1 1 calc R . .
C28 C 0.76448(17) 0.10398(7) 0.92468(15) 0.0133(2) Uani 1 1 d . . .
H28A H 0.6839 0.0659 0.8764 0.016 Uiso 1 1 calc R . .
C29 C 0.96474(18) 0.08068(7) 0.95615(15) 0.0150(2) Uani 1 1 d . . .

loop_
 _atom_site_aniso_label
 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
O1 0.0123(4) 0.0217(5) 0.0176(4) -0.0033(4) 0.0043(3) 0.0016(4)
N1 0.0113(5) 0.0183(5) 0.0135(5) -0.0030(4) 0.0034(4) -0.0002(4)
O2 0.0100(4) 0.0170(5) 0.0270(5) -0.0046(4) 0.0048(4) -0.0019(3)
C2 0.0093(5) 0.0197(6) 0.0126(5) -0.0040(5) 0.0018(4) -0.0002(5)
O3 0.0097(4) 0.0233(5) 0.0225(5) -0.0103(4) 0.0031(4) -0.0017(4)
C3 0.0106(5) 0.0258(7) 0.0171(6) -0.0062(5) 0.0042(4) -0.0043(5)
O4 0.0125(4) 0.0223(5) 0.0186(4) 0.0048(4) 0.0037(3) 0.0018(4)
C4 0.0226(7) 0.0232(7) 0.0239(7) -0.0064(5) 0.0092(6) -0.0084(6)
O5 0.0164(5) 0.0205(5) 0.0434(7) 0.0067(5) 0.0098(5) 0.0049(4)
C5 0.0275(7) 0.0211(7) 0.0241(7) -0.0019(5) 0.0111(6) -0.0058(6)
O6 0.0092(4) 0.0184(5) 0.0277(5) -0.0005(4) 0.0060(4) 0.0001(3)
C6 0.0186(6) 0.0194(6) 0.0189(6) 0.0006(5) 0.0069(5) 0.0014(5)
C7 0.0113(5) 0.0169(6) 0.0149(6) -0.0027(5) 0.0039(4) -0.0021(4)
N8 0.0106(5) 0.0174(5) 0.0145(5) -0.0020(4) 0.0040(4) 0.0007(4)
N9 0.0120(5) 0.0249(6) 0.0194(5) -0.0027(5) 0.0069(4) 0.0004(4)
N10 0.0122(5) 0.0299(6) 0.0197(5) -0.0041(5) 0.0046(4) 0.0004(5)
N11 0.0109(5) 0.0258(6) 0.0181(5) -0.0039(5) 0.0037(4) 0.0005(4)
C12 0.0133(6) 0.0200(6) 0.0168(6) -0.0032(5) 0.0022(5) 0.0018(5)
C13 0.0187(6) 0.0177(6) 0.0163(6) -0.0020(5) 0.0014(5) 0.0004(5)
C14 0.0365(9) 0.0268(8) 0.0274(8) -0.0067(6) 0.0156(7) -0.0089(7)
C15 0.0434(10) 0.0303(9) 0.0349(9) -0.0042(7) 0.0181(8) -0.0126(8)
C16 0.0515(12) 0.0240(8) 0.0314(9) 0.0043(7) 0.0065(8) -0.0092(8)
C17 0.0421(10) 0.0180(7) 0.0316(8) -0.0022(6) 0.0062(7) 0.0006(7)
C18 0.0277(8) 0.0192(7) 0.0313(8) -0.0018(6) 0.0041(6) 0.0044(6)
C19 0.0133(5) 0.0150(6) 0.0131(5) 0.0001(4) 0.0003(4) 0.0001(4)
C20 0.0179(6) 0.0146(6) 0.0121(5) -0.0005(4) 0.0028(4) -0.0007(5)
C21 0.0176(6) 0.0182(7) 0.0278(7) -0.0037(5) 0.0029(5) -0.0002(5)
C22 0.0242(8) 0.0247(8) 0.0353(9) -0.0036(7) 0.0022(7) -0.0080(6)
C23 0.0381(9) 0.0169(7) 0.0327(8) -0.0017(6) 0.0015(7) -0.0085(6)
C24 0.0375(9) 0.0143(7) 0.0359(8) -0.0011(6) 0.0000(7) 0.0040(6)
C25 0.0232(7) 0.0170(6) 0.0256(7) -0.0007(5) 0.0007(6) 0.0034(5)
C26 0.0104(5) 0.0158(6) 0.0133(5) 0.0023(4) 0.0043(4) 0.0008(4)
C27 0.0078(5) 0.0154(6) 0.0147(5) -0.0015(4) 0.0027(4) 0.0001(4)
C28 0.0087(5) 0.0155(6) 0.0152(5) -0.0015(4) 0.0021(4) 0.0002(4)
C29 0.0105(5) 0.0190(6) 0.0156(5) -0.0010(5) 0.0038(4) 0.0017(5)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
```

```
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters are
only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
;

loop_
 _geom_bond_atom_site_label_1
 _geom_bond_atom_site_label_2
 _geom_bond_distance
 _geom_bond_site_symmetry_2
 _geom_bond_publ_flag
O1 C26 1.2532(16) . ?
N1 C6 1.5107(19) . ?
N1 C2 1.5178(17) . ?
N1 H1NB 0.8824 . ?
N1 H1NA 0.8903 . ?
O2 C26 1.2577(16) . ?
C2 C7 1.5116(18) . ?
C2 C3 1.5350(18) . ?
C2 C12 1.548(2) . ?
O3 C27 1.4176(16) . ?
O3 H3O 0.8727 . ?
C3 C4 1.524(2) . ?
C3 H3A 0.9900 . ?
C3 H3B 0.9900 . ?
O4 C28 1.4122(16) . ?
O4 H4O 0.7209 . ?
C4 C5 1.528(2) . ?
C4 H4A 0.9900 . ?
C4 H4B 0.9900 . ?
O5 C29 1.2090(18) . ?
C5 C6 1.521(2) . ?
C5 H5A 0.9900 . ?
C5 H5B 0.9900 . ?
O6 C29 1.3152(17) . ?
O6 H6O 0.8145 . ?
C6 H6A 0.9900 . ?
C6 H6B 0.9900 . ?
C7 N11 1.3231(17) . ?
C7 N8 1.3458(17) . ?
N8 N9 1.3593(15) . ?
N8 C19 1.4713(16) . ?
N9 N10 1.2947(17) . ?
N10 N11 1.3612(17) . ?
C12 C13 1.541(2) . ?
C12 H12B 1.0064 . ?
C12 H12A 0.9556 . ?
C13 C14 1.533(2) . ?
C13 C18 1.538(2) . ?
C13 H13A 1.0000 . ?
```

```
C14 C15 1.539(2) . ?
C14 H14A 0.9900 . ?
C14 H14B 0.9900 . ?
C15 C16 1.528(3) . ?
C15 H15A 0.9900 . ?
C15 H15B 0.9900 . ?
C16 C17 1.526(3) . ?
C16 H16A 0.9900 . ?
C16 H16B 0.9900 . ?
C17 C18 1.533(2) . ?
C17 H17A 0.9900 . ?
C17 H17B 0.9900 . ?
C18 H18A 0.9900 . ?
C18 H18B 0.9900 . ?
C19 C20 1.5132(19) . ?
C19 H19A 0.9900 . ?
C19 H19B 0.9900 . ?
C20 C25 1.392(2) . ?
C20 C21 1.395(2) . ?
C21 C22 1.395(2) . ?
C21 H21A 0.9500 . ?
C22 C23 1.382(3) . ?
C22 H22A 0.9500 . ?
C23 C24 1.386(3) . ?
C23 H23A 0.9500 . ?
C24 C25 1.393(2) . ?
C24 H24A 0.9500 . ?
C25 H25A 0.9500 . ?
C26 C27 1.5335(17) . ?
C27 C28 1.5322(17) . ?
C27 H27A 1.0000 . ?
C28 C29 1.5328(18) . ?
C28 H28A 1.0000 . ?

loop_
 _geom_angle_atom_site_label_1
 _geom_angle_atom_site_label_2
 _geom_angle_atom_site_label_3
 _geom_angle
 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
 _geom_angle_publ_flag
C6 N1 C2 115.90(11) . . ?
C6 N1 H1NB 109.7 . . ?
C2 N1 H1NB 104.4 . . ?
C6 N1 H1NA 108.1 . . ?
C2 N1 H1NA 109.6 . . ?
H1NB N1 H1NA 109.1 . . ?
C7 C2 N1 107.61(10) . . ?
C7 C2 C3 112.21(11) . . ?
N1 C2 C3 106.74(11) . . ?
C7 C2 C12 112.55(11) . . ?
N1 C2 C12 107.85(10) . . ?
C3 C2 C12 109.59(11) . . ?
C27 O3 H3O 106.8 . . ?
C4 C3 C2 113.25(12) . . ?
C4 C3 H3A 108.9 . . ?
```

```
C2 C3 H3A 108.9 . . ?
C4 C3 H3B 108.9 . . ?
C2 C3 H3B 108.9 . . ?
H3A C3 H3B 107.7 . . ?
C28 O4 H4O 108.1 . . ?
C3 C4 C5 109.75(12) . . ?
C3 C4 H4A 109.7 . . ?
C5 C4 H4A 109.7 . . ?
C3 C4 H4B 109.7 . . ?
C5 C4 H4B 109.7 . . ?
H4A C4 H4B 108.2 . . ?
C6 C5 C4 110.99(12) . . ?
C6 C5 H5A 109.4 . . ?
C4 C5 H5A 109.4 . . ?
C6 C5 H5B 109.4 . . ?
C4 C5 H5B 109.4 . . ?
H5A C5 H5B 108.0 . . ?
C29 O6 H6O 112.8 . . ?
N1 C6 C5 110.98(12) . . ?
N1 C6 H6A 109.4 . . ?
C5 C6 H6A 109.4 . . ?
N1 C6 H6B 109.4 . . ?
C5 C6 H6B 109.4 . . ?
H6A C6 H6B 108.0 . . ?
N11 C7 N8 108.42(11) . . ?
N11 C7 C2 125.76(11) . . ?
N8 C7 C2 125.76(11) . . ?
C7 N8 N9 108.00(11) . . ?
C7 N8 C19 133.69(11) . . ?
N9 N8 C19 118.29(11) . . ?
N10 N9 N8 106.69(11) . . ?
N9 N10 N11 110.63(11) . . ?
C7 N11 N10 106.25(11) . . ?
C13 C12 C2 119.88(11) . . ?
C13 C12 H12B 106.5 . . ?
C2 C12 H12B 107.0 . . ?
C13 C12 H12A 108.5 . . ?
C2 C12 H12A 108.0 . . ?
H12B C12 H12A 106.2 . . ?
C14 C13 C18 109.75(13) . . ?
C14 C13 C12 112.97(12) . . ?
C18 C13 C12 108.55(12) . . ?
C14 C13 H13A 108.5 . . ?
C18 C13 H13A 108.5 . . ?
C12 C13 H13A 108.5 . . ?
C13 C14 C15 112.14(13) . . ?
C13 C14 H14A 109.2 . . ?
C15 C14 H14A 109.2 . . ?
C13 C14 H14B 109.2 . . ?
C15 C14 H14B 109.2 . . ?
H14A C14 H14B 107.9 . . ?
C16 C15 C14 111.13(16) . . ?
C16 C15 H15A 109.4 . . ?
C14 C15 H15A 109.4 . . ?
C16 C15 H15B 109.4 . . ?
C14 C15 H15B 109.4 . . ?
H15A C15 H15B 108.0 . . ?
```

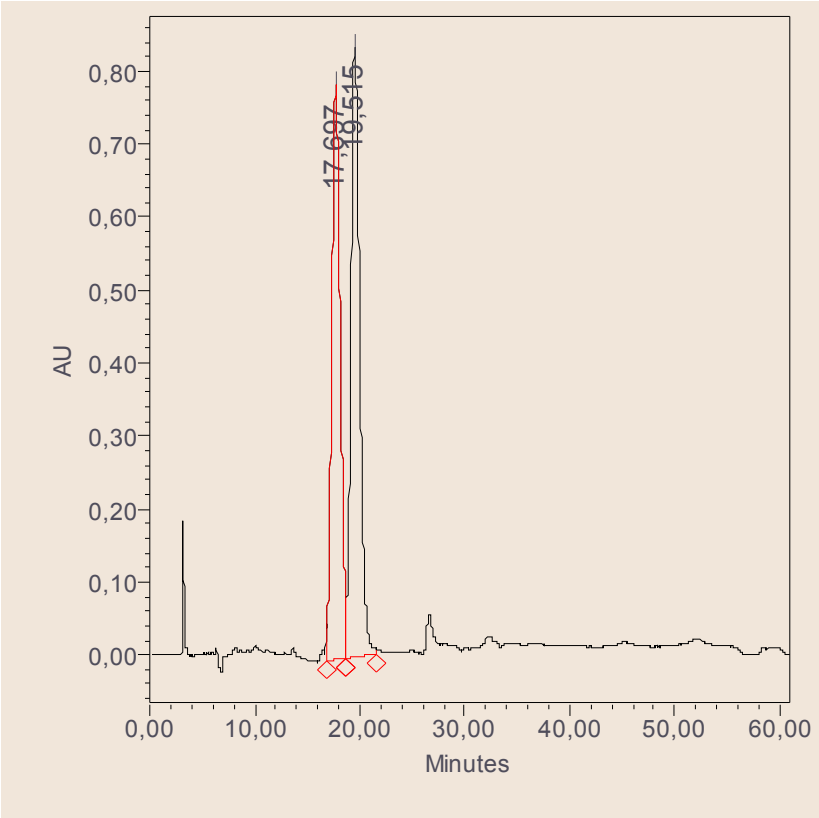
```
C17 C16 C15 110.34(15) . . ?
C17 C16 H16A 109.6 . . ?
C15 C16 H16A 109.6 . . ?
C17 C16 H16B 109.6 . . ?
C15 C16 H16B 109.6 . . ?
H16A C16 H16B 108.1 . . ?
C16 C17 C18 111.15(15) . . ?
C16 C17 H17A 109.4 . . ?
C18 C17 H17A 109.4 . . ?
C16 C17 H17B 109.4 . . ?
C18 C17 H17B 109.4 . . ?
H17A C17 H17B 108.0 . . ?
C17 C18 C13 112.29(14) . . ?
C17 C18 H18A 109.1 . . ?
C13 C18 H18A 109.1 . . ?
C17 C18 H18B 109.1 . . ?
C13 C18 H18B 109.1 . . ?
H18A C18 H18B 107.9 . . ?
N8 C19 C20 113.03(11) . . ?
N8 C19 H19A 109.0 . . ?
C20 C19 H19A 109.0 . . ?
N8 C19 H19B 109.0 . . ?
C20 C19 H19B 109.0 . . ?
H19A C19 H19B 107.8 . . ?
C25 C20 C21 119.56(13) . . ?
C25 C20 C19 121.57(12) . . ?
C21 C20 C19 118.73(12) . . ?
C20 C21 C22 119.96(14) . . ?
C20 C21 H21A 120.0 . . ?
C22 C21 H21A 120.0 . . ?
C23 C22 C21 120.52(15) . . ?
C23 C22 H22A 119.7 . . ?
C21 C22 H22A 119.7 . . ?
C22 C23 C24 119.40(15) . . ?
C22 C23 H23A 120.3 . . ?
C24 C23 H23A 120.3 . . ?
C23 C24 C25 120.81(15) . . ?
C23 C24 H24A 119.6 . . ?
C25 C24 H24A 119.6 . . ?
C20 C25 C24 119.74(15) . . ?
C20 C25 H25A 120.1 . . ?
C24 C25 H25A 120.1 . . ?
O1 C26 O2 127.11(12) . . ?
O1 C26 C27 116.43(11) . . ?
O2 C26 C27 116.45(11) . . ?
O3 C27 C28 109.46(10) . . ?
O3 C27 C26 110.44(10) . . ?
C28 C27 C26 112.06(10) . . ?
O3 C27 H27A 108.3 . . ?
C28 C27 H27A 108.3 . . ?
C26 C27 H27A 108.3 . . ?
O4 C28 C27 111.67(11) . . ?
O4 C28 C29 113.52(11) . . ?
C27 C28 C29 107.61(10) . . ?
O4 C28 H28A 107.9 . . ?
C27 C28 H28A 107.9 . . ?
C29 C28 H28A 107.9 . . ?
```

```
O5 C29 O6 125.87(12) . . ?
O5 C29 C28 121.55(12) . . ?
O6 C29 C28 112.51(12) . . ?

loop_
 _geom_torsion_atom_site_label_1
 _geom_torsion_atom_site_label_2
 _geom_torsion_atom_site_label_3
 _geom_torsion_atom_site_label_4
 _geom_torsion
 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
C6 N1 C2 C7 67.07(14) . . . . ?
C6 N1 C2 C3 -53.57(14) . . . . ?
C6 N1 C2 C12 -171.26(11) . . . . ?
C7 C2 C3 C4 -61.76(15) . . . . ?
N1 C2 C3 C4 55.90(14) . . . . ?
C12 C2 C3 C4 172.44(11) . . . . ?
C2 C3 C4 C5 -59.27(16) . . . . ?
C3 C4 C5 C6 56.02(17) . . . . ?
C2 N1 C6 C5 53.95(15) . . . . ?
C4 C5 C6 N1 -53.01(16) . . . . ?
N1 C2 C7 N11 11.07(19) . . . . ?
C3 C2 C7 N11 128.20(15) . . . . ?
C12 C2 C7 N11 -107.62(15) . . . . ?
N1 C2 C7 N8 -171.93(13) . . . . ?
C3 C2 C7 N8 -54.79(18) . . . . ?
C12 C2 C7 N8 69.39(17) . . . . ?
N11 C7 N8 N9 0.52(16) . . . . ?
C2 C7 N8 N9 -176.92(13) . . . . ?
N11 C7 N8 C19 -177.46(14) . . . . ?
C2 C7 N8 C19 5.1(2) . . . . ?
C7 N8 N9 N10 -0.76(16) . . . . ?
C19 N8 N9 N10 177.59(12) . . . . ?
N8 N9 N10 N11 0.72(17) . . . . ?
N8 C7 N11 N10 -0.09(16) . . . . ?
C2 C7 N11 N10 177.35(13) . . . . ?
N9 N10 N11 C7 -0.40(17) . . . . ?
C7 C2 C12 C13 57.10(15) . . . . ?
N1 C2 C12 C13 -61.45(15) . . . . ?
C3 C2 C12 C13 -177.29(11) . . . . ?
C2 C12 C13 C14 -81.61(16) . . . . ?
C2 C12 C13 C18 156.43(12) . . . . ?
C18 C13 C14 C15 -54.21(19) . . . . ?
C12 C13 C14 C15 -175.50(14) . . . . ?
C13 C14 C15 C16 56.2(2) . . . . ?
C14 C15 C16 C17 -56.4(2) . . . . ?
C15 C16 C17 C18 56.5(2) . . . . ?
C16 C17 C18 C13 -56.3(2) . . . . ?
C14 C13 C18 C17 54.34(18) . . . . ?
C12 C13 C18 C17 178.24(13) . . . . ?
C7 N8 C19 C20 92.60(17) . . . . ?
N9 N8 C19 C20 -85.22(15) . . . . ?
N8 C19 C20 C25 40.20(18) . . . . ?
```

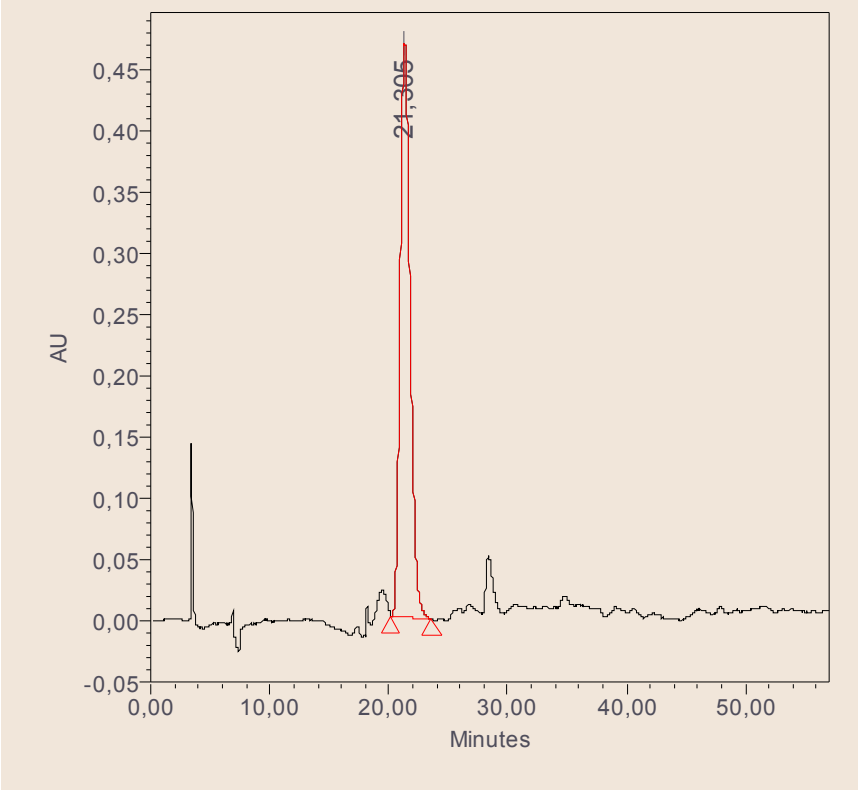
```
N8 C19 C20 C21 -144.10(13) . . . . ?
C25 C20 C21 C22 -0.1(2) . . . . ?
C19 C20 C21 C22 -175.91(14) . . . . ?
C20 C21 C22 C23 0.3(3) . . . . ?
C21 C22 C23 C24 -0.2(3) . . . . ?
C22 C23 C24 C25 0.0(3) . . . . ?
C21 C20 C25 C24 -0.1(2) . . . . ?
C19 C20 C25 C24 175.52(15) . . . . ?
C23 C24 C25 C20 0.2(3) . . . . ?
O1 C26 C27 O3 -14.68(16) . . . . ?
O2 C26 C27 O3 166.43(11) . . . . ?
O1 C26 C27 C28 -137.00(12) . . . . ?
O2 C26 C27 C28 44.12(16) . . . . ?
O3 C27 C28 O4 -60.92(13) . . . . ?
C26 C27 C28 O4 61.95(14) . . . . ?
O3 C27 C28 C29 64.30(13) . . . . ?
C26 C27 C28 C29 -172.82(11) . . . . ?
O4 C28 C29 O5 -153.74(13) . . . . ?
C27 C28 C29 O5 82.15(16) . . . . ?
O4 C28 C29 O6 29.08(16) . . . . ?
C27 C28 C29 O6 -95.03(13) . . . . ?

_diffrn_measured_fraction_theta_max    1.000
_diffrn_reflns_theta_full              30.00
_diffrn_measured_fraction_theta_full   1.000
_refine_diff_density_max    0.282
_refine_diff_density_min   -0.168
_refine_diff_density_rms    0.037

_publ_section_references
;
Bruker, Programs APEX II, version 2.0-1; SAINT, version 7.23A;
SADABS, version 2004/1; XPREP, version 2005/2;
SHELXTL, version 6.1. Bruker AXS Inc., Madison, WI, USA, 2005.

G.M. Sheldrick, Programs SHELXS97 (crystal structure solution)
and SHELXL97 (crystal structure refinement), University  of
Gottingen, Germany, 1997.
;
```

3. HPLC analysis for 13 a and 13b

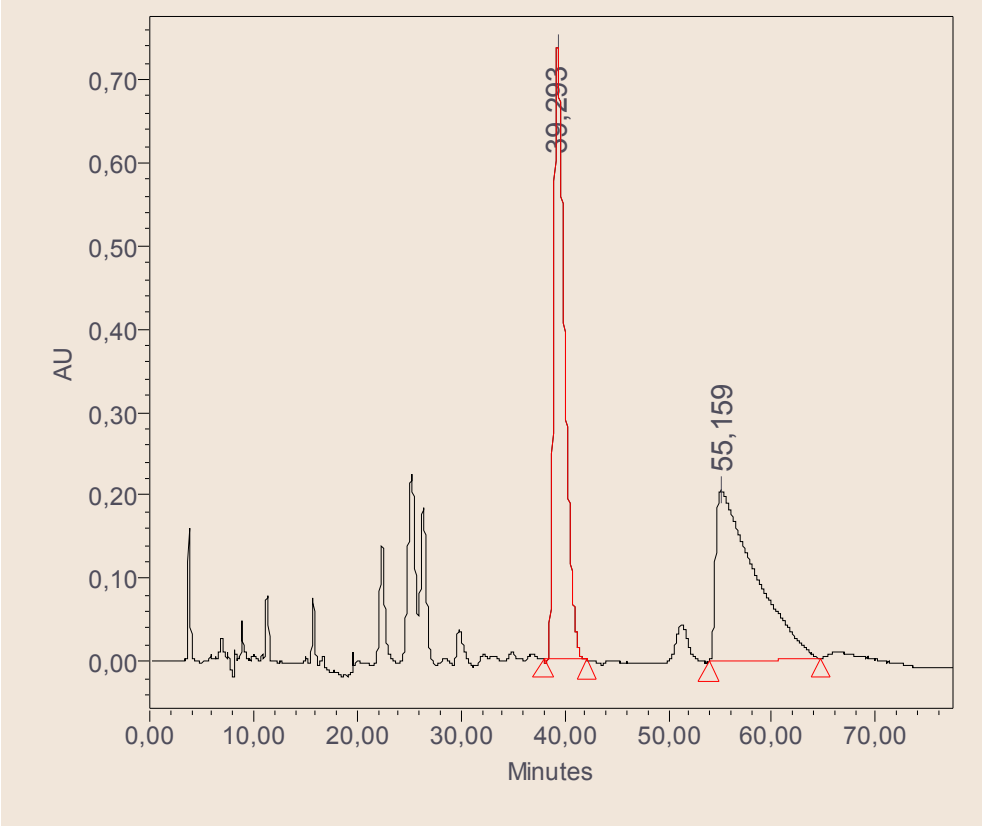
13a - Racemic mixture



13a – enantiomerically enriched compound



13b - Racemic mixture



13b – enantiomerically enriched compound